REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN PART II

VOLUME 2 - APPENDICES REVISION: 2

ENVIRO-CHEM SITE ZIONSVILLE, INDIANA

REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN PART II - QUALITY ASSURANCE PROJECT PLAN VOLUME 2 - APPENDICES

ENVIRO-CHEM SITE ZIONSVILLE, INDIANA

MARCH 24, 1992

REVISION: 2

PREPARED FOR:

ENVIRO-CHEM TRUSTEES

PREPARED BY:

ENVIRONMENTAL RESOURCES MANAGEMENT-NORTH CENTRAL, INC.
102 WILMOT ROAD, SUITE 300
DEERFIELD, ILLINOIS 60015
PROJECT NO. 91104

APPENDIX A

COMPUCHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURES AND OTHER INFORMATION

APPENDIX A

COMPUCHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURES AND OTHER INFORMATION

TABLE OF CONTENTS

A.1	Warzyn Engineering, Inc Standard Operating Procedure for Tin Analysis
A.2	Standard Operating Procedure for 1,1-Dichloroethane Analysis
A.3	Chain-of-Custody Procedures
A.4	Standard Operating Procedures for Production Planning and Control
A.5	Data Processing Procedures
A.6	Corrective Action and Performance Audits Procedures
A.7	Facilities, Equipment, and Services
A.8	SOP Modifications and Special Considerations
A.9	Standard Operating Procedure for Volatile Organics in Soil Analysis

APPENDIX A.1

WARZYN ENGINEERING, INC.

STANDARD OPERATING PROCEDURE FOR TIN ANALYSIS

APPENDIX A.1

WARZYN ENGINEERING, INC.

STANDARD OPERATING PROCEDURES AND OTHER INFORMATION

TABLE OF CONTENTS

Part I: Acid Digestion for Aqueous Samples and Extracts

Part II: Inductively Coupled Plasma - Atomic Emission Spectrometric Method

Part III: Additional Information

Effective: 7-2-91

ACID DIGESTION FOR AQUEOUS SAMPLES AND EXTRACTS ICP/Flame-AA

Scope and Application:

This acid digestion is applicable to all aqueous sample matrices. A nitric/hydrochloric acid digestion is used to prepare all samples which are to be analyzed by flame atomic absorption spectroscopy (flame-AA) or by inductively coupled plasma spectroscopy (ICP). A nitric acid/hydrogen peroxide digestion is used to prepare samples for analysis by graphite furnace atomic absorption spectroscopy (GFAA).

Method: Nitric acid/hydrogen peroxide and nitric/hydrochloric acid digestions

Reference: "Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Sample Handling: Aqueous samples must be acidified with concentrated nitric acid to pH

< 2. Set up digestion as soon as possible; digested sample must be analyzed within 6 months.

Reagents and Apparatus:

1. Hot plate

2. 250 mL beakers

- 3. 100 mL graduated cylinders
- 4. Class A volumetric glassware
- 5. Deionized (D.I.) water
- 6. Instra-analyzed nitric acid, or equivalent
- 7. Distilled nitric acid (GFAA digestion only)
- 8. Instra-analyzed HCl acid, or equivalent
- 9. Stock and standard metal solutions
- 10. Whatman #42 filter paper
- 11. Glass or plastic funnels
- 12. Watch glasses
- 13. 30% Hydrogen peroxide

Reagent Preparation:

- 1. <u>Intermediate and working metal solutions:</u> Refer to the specific metal SOP for instructions on preparation.
- 2. <u>1:1 Hydrochloric acid (HCl):</u> Using a graduated cylinder, add 250 mL D.I. water to a to a 500 mL (or 1 L) repipettor. Carefully add 250 mL of concentrated HCl and mix.
- 3. <u>1:1 Nitric acid (HNO3):</u> Using a graduated cylinder, add 250 mL D.I. water to a to a 500 mL (or 1 L) repipettor. Carefully add 250 mL of concentrated HNO3 and mix.

Notes:

- 1. A separate digestion is required for mercury analyzed by the AA-Cold Vapor technique. (See "Mercury Digestion-Aqueous Samples")
- 2. All samples, duplicates, and spikes, as well as any required prep or digested blanks and standards, must be carried through the digestion procedure.
- 3. If samples boil or go to dryness (any dry spots on the bottom of the beaker) at any time during the digestion, some of the analyte may have been lost. The digestion must be discarded and the affected samples must be reprepared.
- 4. If elevated analyte levels are expected, the spike concentration may be increased accordingly.

Procedure:

Digestion Procedure for Flame-AA and ICP:

- 1. All glassware must be acid-washed with 1:1 nitric acid and thoroughly rinsed with D.I. water prior to use.
- 2. Measure out 100 mL aliquots of samples, blanks, and standards into 250 mL beakers using a graduated cylinder.
- 3. Add 2.0 mL of 1:1 HNO₃ and 10 mL of 1:1 HCl.
- 4. Cover with a watch glass and heat on the hot plate for 2 hours or until the volume has been reduced to between 25 and 50 mL. Adjust the temperature of the hot plate as needed to prevent samples from boiling.
- 5. Allow samples to cool. If any insoluble material remains, filter samples through Whatman #42 filters. Quantitatively transfer digested samples, blanks, and standards into 100 mL volumetric flasks. Rinse beakers and filters with D.I. water and dilute to volume to 100 mL.
- 6. Samples are now ready for analysis using the AA-flame or ICP methods.

Digestion Procedure for GFAA:

- 1. All glassware must be acid-washed with 1:1 nitric acid and thoroughly rinsed with D.I. water prior to use.
- 2. Measure out 100 mL aliquots of samples, blanks, and standards into 250 mL beakers using a graduated cylinder.
- 3. Add 1.0 mL of 1:1 HNO₃ and 2.0 mL of 30% H₂O₂.
- 4. Cover with a watch glass and heat on the hot plate for 2 hours or until the volume has been reduced to between 25 and 50 mL. Adjust the temperature of the hot plate as needed to prevent samples from boiling.

- 5. Allow samples to cool. If any insoluble material remains, filter samples through Whatman #42 filters. Quantitatively transfer digested samples, blanks, and standards into 100 mL volumetric flasks. Rinse beakers and filters with D.I. water and dilute to volume to 100 mL.
- 6. Samples are now ready for GFAA analysis.

Quality Control:

- 1. A digested blank and standard (spiked blank) must be included with each batch of samples that is digested. The blank is a check for possible contamination during the digestion process; the standard is a check for possible analyte loss during digestion.
- 2. A matrix spike and duplicate must be prepared, at a minimum, for every 10 samples digested. If fewer than 10 samples are digested a spike and duplicate are still required.

INDUCTIVELY COUPLED PLASMA - ATOMIC EMISSION SPECTROMETRIC METHOD *

Scope and Application: Metals in solution can be readily analyzed by atomic emission using an inductively coupled plasma. Dissolved metals are determined in Total metals are determined in filtered and acidified samples. acidified, but unfiltered samples. Appropriate steps must be taken in all analyses to ensure that potential spectral interferences are taken into account.

Method: Inductively coupled plasma - atomic emission.

Reference: "Methods for Chemical Analysis of Water and Wastes", Method 200.7 EPA

> "Inductively Coupled Plasma - Atomic Emission Spectroscopy", Method 6010, SW-846, November 1986,

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

"Instructions: Plasma 40 Emission Spectrometer", Perkin-Elmer, 1987.

Sample Handling:

Acidify aqueous samples with concentrated nitric acid to pH <2. All samples must be digested prior to analysis (refer to appropriate digestion procedure). All samples must be analyzed within 6 months of sampling date.

Reagents and Appartus:

Plasma 40 Perkin-Elmer ICP Spectrometer

Argon (liquid: "high purity" or gaseous: "prepurified" grade)

Stock and intermediate metal standard solutions EPA, ERA, or other reference standard solutions

5. Nitric acid, conc. (instra-analyzed or equivalent grade)

6. Class A volumetric glassware

Deionized water

Disposable 15 mL centrifuge tubes

100 uL Eppendorf pipetter

10. 5 or 10 mL Oxford pipetter
11. Yttrium or Scandium stock solution
12. IBM AT Computer or equivalent

13. Epson 800 printer

Procedure:

Instrument Set-Up Procedure for Plasma 40:

Turn ON power switch if necessary (routinely left ON throughout week). Allow 1 hour for RF generator to warm up and electronic and optical components to achieve thermal equilibrium.

*Note: Only the results of the analysis for tin will be reported for this project.

[METCONT-199]

- 2. Perform daily maintenance as specified in Maintenance Procedures: check pump, pump tubing, and nebulizer tips for wear, cleanliness, etc.
- 3. Turn on argon at tank. The first three indicator lights on the ICP (Power, RF ready, Interlock) should light.
- 4. Lock pump tubing in place, raise torch to the "ignite" position, and press "RF on".
- 5. When plasma ignites, lower torch to the run position (the injector tip should be even with or just below the bottom of the lowest RF coil).
- 6. Turn on pump and aspirate rinse water*. Allow plasma to stabilize 30 to 40 minutes before starting analysis.

Computer Start-Up Procedure:

- 1. Turn computer and printer power on (the computer will automatically start with a memory check).
- 2. Type CD ICP and press Return to enter the ICP directory. Then type ICP and press Return again to load software (approximately 10-15 seconds).
- 3. Perform a BEC check as specified in Maintenance Procedures. The BEC and CV values must be within the specified range before any analysis is done.

Sample Analysis:

- 1. Before starting analysis, for each element to be analyzed:
 - a. Press F1 to select the Element mode, type the appropriate element file name and press Alt F9 to retrieve it from Library.
 - b. Press F8 to select Spectrum mode.
 - c. Analyze a single element standard at approximately 2-10X the IDL.
 - d. Analyze the ICS AB solution.
 - e. Analyze 1-3 samples representative of the digestion set.
 - f. Compare the displayed spectra to check for spectral interferences. Reset background correction points as needed. If there are overlapping peaks or other spectral interferences present, an alternate wavelength or interelement correction must be used.
 - g. Press F8 to leave the Spectrum mode. If wavelength calibration or background correction points were changed, press F9 to save the changes.
- Rinse water should be D.I. water with a small amount of liquid detergent (such as Liquinox or Whisk) added to improve wetting of tubing and spray chamber. Approximately 1-2 mL of soap per 500 mL water should be sufficient.

- 2. To store a list of sample labels to be used for the analytical run, select Report mode (F3), then ID/Wt mode (F8). Enter a file name, type in N (no) in the field for raw emission counts, mg/L for uncorrected units, and leave the corrected units field blank. Enter sample labels in the sample ID field in the exact order of analysis; include all check standards, QC samples, etc. If it is possible that additional samples may be added to the end of an analytical run (dilutions, post-digestion spikes, linear range standards, etc.) add additional sample labels to the ID/Wt file in the form of single letters (A, B, C, etc.) and manually write in the correct sample labels after the analytical run is completed. Alternatively, a new ID/Wt file may be created after the analytical run is completed (in this case the raw data must be reprinted with the new file by selecting Report Format 1 (F5) in the report mode and responding to the prompts). Save ID/Wt files by pressing F9 (to library).
- 3. Press F2 to select the Method mode.
- 4. Type the method file name and press Alt F9 to retrieve the desired method panel from Library, or create a new panel using existing element files. Standard conditions are 35 second read delay, 2 replicates per sample, report format #2 and a data file name composed of the date (mmdd) and a sequential letter identifier (e.g. 0123B for the second analytical run on Jan 23). An internal standard (usually yttrium) must be included in any method. Background correction points are already included in each element file.
- 5. Add yttrium (or scandium) stock solution (1000 mg/L) as an internal standard to all standards, blanks, and samples in a ratio of 0.1 mL yttrium stock to 10 mL sample. This allows automatic correction for matrix differences in viscosity, surface tension, etc.. If the autosampler is to be used, samples can be pipetted directly into 15 mL centrifuge tubes. Otherwise mix sample and yttrium in small disposable beakers.

If autosampler is used: *

- 1. If the autosampler is to be used, load sampler starting with the calibration standards in order of decreasing concentration (highest concentration first, calibration blank last).
- 2. Start automatic run (F5). Respond to the prompts that appear at the bottom of the screen:
 - a. "Press start function key to begin this analysis": press F5
 - b. "Enter ID/Wt file": type ID/Wt file name and press Return.
 - c. "Do you wish to rinse between tubes (Y or N)": type Y and press Return. N may be selected only for clean samples where no carry-over problems are anticipated. Always rinse between samples when analysis is following CLP protocols, or analyzing for Sb, Cr, or Zn.
- * Note: The ICP autosampler will be used for all analytical runs except in the event of mechanical failure.

- d. "Enter position of the last sample in tray": type appropriate number and press
 Return (you may wish to enter a number several positions past the last sample to
 allow room for the addition of necessary dilutions, etc. at the end of the run).
- e. "Do you wish to re-standardize (Y or N)": type N or Y and press Return. N is usually selected. Y will allow restandardization of the instrument during an automatic run but additional autosampler positions will be unavailable for samples. If Y is selected, additional on-screen instructions will prompt for position of additional calibration standards.
- f. "Do you wish to wavelength calibrate during the analysis? (Y or N)": type N or Y and press Return. N is usually selected. Y will allow recalibration of all wavelengths used in the current method before analysis is started but additional autosampler positions will be unavailable for samples. If Y is selected, additional on-screen instructions will prompt for position of additional wavelength calibration standards. The system will then begin the analysis.
- 3. When the analysis is complete press F2 to select Method mode before exiting software to ensure the data file is stored permanently. Then set up the next panel, return to Report mode to set up a new ID/Wt file or reprint data, or press ESC to exit the ICP software.

If samples are to be run manually:

- 1. Press F2 to start a manual run and respond to prompts to calibrate instrument: Press F6 (Standard), aspirate the first calibration standard and press Return. At the prompt, aspirate the next standard(s) and press Return. When all calibration standards have been analyzed press F5 (Blank) aspirate the calibration blank and press Return. This completes the instrument calibration.
- 2. To analyze samples, type in sample label if needed, press F7 (Sample), aspirate sample and press Return. Repeat with all samples in the run.

Computer Shut-Down Procedure:

- 1. When analysis is complete press F2 (Method mode). At the message "Do you wish to quit method"? Type Y.
- 2. Press "ESC". At the message "Do you wish to quit method?" Type Y.
- 3. Turn off computer power switch.
- 4. Turn off printer.

Caution: Never turn off computer power while still using ICP software. This can cause partial loss of files and other errors.

Instrument Shut-Down Procedure:

1. Aspirate a dilute nitric acid solution (approx. 10%) for 1 to 2 minutes to clean sample introduction system.

- 2. Aspirate D.I. water for 5 minutes to rinse system thoroughly.
- 3. Turn off pump and release pump tubing.
- 4. Press "RF off" to extinguish plasma.
- 5. Shut off argon flow at tank.
- 6. If the ICP will not be used for 2 days or more, turn off ICP power switch. Otherwise, leave the ICP power ON.

Quality Control:

- 1. Establish a standard curve with the appropriate calibration standards plus a blank. Record the emission count for the internal standard in the ICP log book. The emission count should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check pump tubing, nebulizer tips, nebulizer flow, wavelength calibration, etc.).
- 2. The first analyses for each analytical run are, in order:
 - a. Initial calibration verification standard (ICV)
 - b. Initial calibration blank (ICB)
 - c. Initial standard at 2X the CRDL (CRI). Note: The CRI is not necessary for Ca, Mg, Na or K.
 - d. Initial interference check sample, solution A (ICSA).
 - e. Initial interference check sample, solution AB (ICSAB)
 - f. Laboratory control standard an ERA, EPA, or other reference standard digested with the sample set (LCS)

To continue with sample analyses, the ICV must be within 90-110% of the true value, the ICB must be less than the CRDL, and the LCS and ICS solutions must be within 80-120% of the true value. If these QC criteria are not met, discontinue the analytical run and perform necessary troubleshooting.

- 3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are to be analyzed, a duplicate and spike are still required. Duplicates and spikes are to be within required control limits or the data must be flagged appropriately (N for spikes, * for duplicates). Additionally, if a digested spike is outside required control limits, a post-digestion spike must be analyzed for that sample.
- 4. For each sample batch (same matrix and project) one sample must be analyzed at an additional 5X dilution for the ICP serial dilution analysis (L). If the original sample concentration is at least 50X above the IDL, the serial dilution must agree within 10% of the original sample concentration or data for all associated samples must be flagged appropriately (E).

- A continuing calibration verification standard (CCV) and blank (CCB) are to be analyzed, at a minimum, after every 10 analyses. If less than 10 analyses are performed, a CCV and CCB are still required. The last samples analyzed in the run are to be a CCV and CCB. The CCVs must be within 90 - 110% of the true value or the samples run after the last acceptable calibration standard are to be reanalyzed.
- At the end of each analytical run, but before the final CCV and CCB, the CRI, and ICS solutions A and AB are to be reanalyzed. The ICS must be within 80-120% of the true value or the samples run after the last acceptable calibration verification standard are to be reanalyzed.
- Refer to the appropriate Quality Assurance Project Plan (QAPP) for project specific QC information (additional QC requirements, matrix spike and duplicate control limits, etc.).
- Detection limit verifications and linear range analyses must be performed each Interelement correction factors are to be determined annually at a minimum. Interelement correction factors must be recalculated on an analyte and wavelength specific basis any time background correction points are changed in an element file. Additionally, for greatest accuracy, interelement correction factors should be re-determined for any analytical batch that is expected to have high concentrations of common interferents (e.g. any soil, sediment, sludge, or leachate matrix).

Daily Maintenance Procedures - Plasma 40

- Pump rollers: With the pump on, feel along the bottom of the pump to determine that all the rollers are turning smoothly with no resistance or pulling. If a "sticky" roller is found a service call must be placed to Perkin-Elmer to correct the problem. A sticky roller will cause rapid deterioration of pump tubing resulting in erratic results.
- Pump (ubing: Check pump tubing for excessive stretching, soft or flattened spots. This can cause irregular or diminished sample flow resulting in reduced sensitivity and lack of precision in sample results. When pump tubing is changed, (usually after 6-8 hours of use) it is necessary to trim ends of the new tubing so the length from the black stops to the end of the tubing is kept constant. Failure to trim tubing ends can cause imprecise results due to a longer sample read delay.
- Nebulizer tips: Remove nebulizer end cap and check nebulizer tips visually and with the cleaning wire for clogs, salt build-up or other deposits. Follow the instructions in the Plasma 40 operating instructions for replacing nebulizer tips if necessary (Part 2, pg 3-8). Used tips may be cleaned by soaking overnight in 10% nitric acid followed by thorough rinsing with D.I. water. Finally, with the argon on, aspirate water and observe the spray pattern. The nebulizer should produce a fine, even mist with no large droplets with the direction of the spray approximately perpendicular to the face of the end cap (should not deviate more than about 20°). If the spray pattern looks uneven, "bent", or is pulsing excessively, recheck pump tubing and review nebulizer maintenance to correct the problem.

- 4. BEC check: This is an indication of how well the ICP system is performing. After the plasma has been ignited and allowed to stabilize for 30-40 minutes perform the following steps:
 - a. At the DOS prompt type ICP and press Return to load software.
 - b. Type MnBEC and press Alt F9 to retrieve this method from Library. Press F6 to start a manual run.
 - c. Press F6 again (standards), aspirate a 1.0 mg/L Mn standard and press Return. The ICP will analyze 10 replicates of this standard. The Coefficient of Variance (CV) for these readings should be <2.0. If a higher values is obtained a sample introduction or instrument calibration problem is indicated. Check pump tubing and wavelength calibration for Mn and repeat the analysis. Record the CV in the maintenance log book.
 - d. Press F5 (blank), aspirate a blank, and press Return to complete the calibration.
 - f. Turn off the torch (RF off), aspirate D.I. water, and press F7 (sample). The resulting concentration should be < |0.040|. If a higher value is obtained, a problem with the sample introduction system is indicated. Review maintenance and, if the problem cannot be corrected, place a service call with Perkin-Elmer. Record the BEC in the maintenance log book.
 - i. Re-light the torch and press ESC to end the manual run. Allow the plasma to stabilize 10-15 minutes before beginning any analysis.
- 5. Final rinse: When analysis for the day is complete, aspirate dilute (approx. 10%) nitric acid for one or two minutes followed by D.I. water for approximately 5 minutes. This will help prevent deposits from building up in the sample introduction system. Remember to release pump tubing when completed.

Weekly Computer Backup:

- 1. Once a week data files should be copied to floppy disks and deleted from the hard disk. Data files on floppy disks should be saved for one year.
- 2. Periodically (every 1-3 months depending on work volume), files should be reviewed, old files deleted and the entire system backed-up.

Other Maintenance:

1. Occasionally, additional maintenance will be necessary to correct problems arising from time and wear on the system. Any additional maintenance performed (including P.E. service calls) should be listed in the maintenance logs. These include periodic cleaning of the torch assembly, inspection of O-rings in torch assembly, and wavelength recalibration. Generally, these procedures will only be performed in response to observed problems. Refer to the Plasma 40 operating manual for specific directions.

ICAP CALIBRATION STANDARDS

Element	Wave- length	Detection Limit(ug/L)	Cal. Std. 1 (ug/L)	Cal. Std. 2 (ug/L)	Cal. Std. 3 (ug/L)	ICV (ug/L)	CCV (ug/L)
Al	237.335	50	20,000	400	•••	2500	4000
Al	396.152	50	20,000	400	•••	2500	4000
Sb	206.833	50	2000	500	250	1000	1000
Ba	233,527	10	10,000	200	•••	500	2000
Ве	313.107	5	1000	20		250	200
Cd	228.802	5	1000	50		500	200
Cd	214.438	5	1000	50	a##	500	200
Ca	317.933	1000	200,000	10,000		10,000	80,000
Cr	267.716	10	10,000	500	200	1000	2000
Cr	205.552	10	10,000	500	200	1000	2000
Co	238.892	50	10,000	200		2500	2000
Co	228.616	10	10,000	200		2500	2000
Cu	324,754	10	10,000	100	•••	1000	2000
Cu	224.700	20	10,000	100	•••	1000	2000
Fe	238.204	20	20,000	200		1000	4000
Pb	220.353	100	10,000	500		5000	2000
Pb	216.999	100	10,000	500	•••	5000	2000
Mg	285.213	1000	100,000	5000	-44	10,000	40,000
Mn	257.610	10	10,000	100		500	2000
Ni	352.454	20	10,000	100		1000	2000
Ni	232.003	20	10,000	100	404	1000	2000
Ag	338.289	10	1000		50	500	200
Na	330.237	2000	100,000	5000	240	20,000	40,000
Sn	189.989	200	10,000	1000	***	2500	5000
V	292.402	50	10,000	500	250	2500	2000
Zn	213.856	10	10,000	100	50	500	2000

[METALS-549]

ICAP-1

80.9 MASS:01 1991 P.08

ICAP CALIBRATION STANDARDS

ICAP calibration standards are prepared from both multi-element stock solutions purchased from SPEX Industries (custom mixed standards) and single element stock solutions from VWR and Baxter (Ricca or Mallinckrodt as available).

XWE-1	XWE-2	XWE-3a	XWE-4a
2000 mg/L Fe 1000 mg/L Cu	20,000 mg/L Ca 10,000 mg/L Mg	1000 mg/L Cr 1000 mg/L Pb	2000 mg/L Al 1000 mg/L Ba
1000 mg/L Mn 1000 mg/L Ni 1000 mg/L Zn	10,000 mg/L Na	1000 mg/L V 100 mg/L Cd	1000 mg/L Co 100 mg/L Be 100 mg/L Ag
XWE-6a		Single Element Stock Solutions 1000 mg/L	
500 mg/L Pb 250 mg/L Co, Al 100 mg/L Cu, Ni, 50 mg/L Ba, Cd,		Sb Ag Be Na Ca V Cr Zn Mg Sn	

Calibration Standard #1:

- 1. For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, Ag, Na, V, Zn: Into a 1 L volumetric flask, add 500 mL of de-ionized (D.I.) water and 50 mL of concentrated HCl. Pipet 10 mL each of XWE-1, XWE-2, XWE-3a, and XWE-4a. Dilute to volume with D.I. water.
- 2. For Sb: Into a 500 mL volumetric flask, add 250 mL of D.I. water and 25 mL of concentrated HCl. Pipet 1.0 mL of 1000 mg/L Sb stock solution. Dilute to volume with D.I. water.
- 3. For Sn: Into a 500 mL volumetric flask, add 250 mL of D.I. water and 25 mL of concentrated HCl. Pipet 5.0 mL of 1000 mg/L Sn stock solution. Dilute to volume with D.I. water.

Calibration Standard #2:

1. For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Nl, Na, V, and Zn: First prepare 10X dilutions each of XWE-1, XWE-2, XWE-3a, and XWE-4a. Then, into a 1 L volumetric flask, add 500 mL of D.I. water and 50 mL of concentrated HCl. Pipet 1.0 mL of XWE-1 (10X dilution), 5.0 mL of XWE-2 (10X dilution), 5.0 mL of XWE-3a (10X dilution), and 2.0 mL of XWE-4a (10X dilution). Dilute to volume with D.I. water.

[METALS-549]

ICAP-2

- 2. For Sb: Into a 200 mL volumetric flask, add 100 mL of D.I. water and 10 mL of concentrated HCl. Pipet 50 mL of Sb Calibration Standard #1 and dilute to volume with D.I. water.
- 3. For Sn: Into a 200 mL volumetric flask, add 100 mL of D.I. water and 10 mL of concentrated HCl. Pipet 20 mL of Sn Calibration Standard #1 and dilute to volume with D.I. water.

Calibration Standard #3:

1. For Cr, Ag, V and Zn: First prepare intermediates as follows:

50 mg/L Ag and Zn: Into a 100 mL volumetric flask, add 10 mL of 1:1 HCl. Pipet 5.0 mL each of single element Ag and Zn stock solutions and dilute to volume with D.I. water.

100 mg/L Cr: Into a 100 mL volumetric flask pipet 10.0 mL of single element Cr stock solution. Add 5 mL of 1:1 HCl and dilute to volume with D.I. water.

50.0 mg/L V: Into a 100 mL volumetric flask pipet 5.0 mL of single element V stock solution. Add 5 mL of 1:1 HCl and dilute to volume with D.I. water.

Then, into a 500 mL volumetric flask, add 250 mL D.I. water and 25 mL of concentrated HCl. Pipet 1.0 mL of 100 mg/L Cr intermediate, 0.5 mL of 10 mg/L Ag-Zn mixed intermediate, and 2.5 of 50 mg/L V intermediate. Dilute to volume with D.I. water.

2. For Sb: Into a 100 mL volumetric flask, add 50 mL of D.I. water and 10 mL of 1:1 HCl. Pipet 50 mL of Calibration Standard #2 and dilute to volume with D.I. water.

Initial Calibration Verification:

- 1. For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Ph, Mg, Mn, Ni, Ag, Na, V, and Zn: Into a 1 L volumetric flask, add 500 mL of D.I. water and 50 mL of concentrated HCl. Pipet 10 mL of XWE-6a, 10 mL of 1000 mg/L Mg stock, 10 mL of 1000 mg/L Ca stock, and 20 mL of 1000 mg/L Na stock solutions. Dilute to volume with D.I. water.
- 2. For Sb: Into a 500 mL volumetric flask, add 250 mL of D.I. water and 25 mL of concentrated HCl. Pipet 0.5 mL of 1000 mg/L Sb stock and dilute to volume with D.I. water.
- 3. For Sn: Into a 1 L volumetric flask, add 500 mL of D.I. water and 50 mL of concentrated HCl. Pipet 2.5 mL of 1000 mg/L Sn stock and dilute to volume with D.I. water.

[METALS-549]

10:26AM P.10

Continuing Calibration Verification Standard:

- 1. For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Ph, Mg, Mn, Ni, Ag, Na, V, and Zn: Into a 1 L volumetric flask, add 500 mL D.I. water and 50 mL of concentrated HCl. Pipet 2.0 mL each of XWE-1, XWE-3a, XWE-4a, and 4.0 mL of XWE-2. Dilute to volume with D.I. water.
- 2. For Sb: Use Sb Initial Calibration Verification Standard.
- 3. For Sn: Into a 200 mL volumetric flask, add 100 mL D.I. water and 10 mL of concentrated HCl. Pipet 1.0 mL of 1000 mg/L Sn stock and dilute to volume with D.I. water.

[METALS-549]

Part III: Additional Information

- 1. Working Linear Range: The working linear range is specific to each element and wavelength. Linear ranges are determined quarterly, at a minimum, and any time method or calibration parameters are changed. Linear range information is reported on Form 12 of the final data package.
- 2. Interferences and Corrective Actions: The possible presence of spectral interferences (including interelement interferences) is evaluated for each batch of samples ("batch" refers to samples of the same project, matrix, and digestion set) and corrected as outlined in the Sample Analysis section of Part II. The presence and extent of other matrix and/or chemical interferences is checked through the required matrix-specific quality control samples (spikes, duplicates, serial dilutions). Any affected data is handled as outlined in the 3/90 SOW, including flagging of data, redigestion of samples, and/or reanalysis as appropriate.
- 3. Safety Precautions: Safety precautions are discussed in detail in the laboratories' Health and Safety Manual and in their Basic Laboratory Requirements.
- 4. Storage of Reagents and Standards: All reagents and standard solutions are stored at room temperature. Stock solutions are kept for no longer than one year. While there are no set guidelines for the shelf life of mixed-element standards, these standards are prepared in quantities sufficient for approximately two months' use. No standards are kept for longer than one year. Alternate source initial calibration standards and laboratory control standards are required for each analytical run and insure that calibration standard solutions are accurate and stable.
- 5. Data Treatment: ICP data is reduced automatically by the ICP operating and reporting software which runs on a dedicated Northgate PC (IBM compatible). Data is simultaneously printed and stored to disk during the analytical run. Raw data includes analyst initials, lab sample number, analyte, date and time of analysis, analyte

concentration in mg/L (for both replicate instrument readings), and the average concentration, standard deviation, and coefficient of variance for each sample. Final reports are generated from raw ICP data files which are downloaded to a Telecation software package. This database and report package includes all algorithms necessary to convert the raw data to final reportables required under the SOW.

- 6. Data Deliverables: Data deliverables will include the following:
 - o Laboratory case narrative,
 - o Forms 1-14 as required under the 3/90 SOW,
 - o All laboratory digestion/sample preparation logs,
 - o Computer printouts of all raw data, and
 - o Copies of all chain-of-custody records.
- 7. Quality Control Requirements: The QC requirements of the 3/90 SOW will be used for this project. These requirements are as follows:
 - o 75-125% recovery for matrix spikes, except where the sample concentration is greater than 4 times the spiking level;
 - o 20% relative percent difference for duplicate analyses, where sample concentration is greater than 5 times the CRDL; and
 - ± CRDL for duplicate analyses where sample concentration is less
 than 5 times the CRDL.

APPENDIX A.2

COMPUCHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURE FOR 1,1-DICHLOROETHANE ANALYSIS

INTRODUCTION TO THE STANDARD OPERATING PROCEDURE FOR ANALYSIS OF 1,1-DICHLOROETHANE IN WATER

CompuChem Laboratories' SOP for the analysis of 1,1-Dichlorocthane (1,1-DCA) in water is included in this Appendix. The following is some additional project-specific information regarding these SOPs.

- 1. Although the only parameter to be reported from this analysis is 1,1-DCA, the SOP will be followed exactly with respect to the standards used for calibration and spiking.
- 2. The complete acceptance criteria for MS/MSD samples is presented in "Attachment 7," at the end of the SOP.
- 3. The list of deliverables is attached at the end of the SOP.

Standard Operating Procedure (SOP) Documentation Form

Standard Operating Procedures (SOPs) describe in detail how tasks are performed in specific areas. Because they are used for training as well as for legal documentation, it is important that SOPs reflect the most current practices of the laboratory or department. In turn, we must keep careful records of who wrote or revised SOPs, when they became effective, and when it is time for SOPs to be reviewed. This form must accompany all SOPs to help us record that information.

Please be sure that the shaded area of this form is completed before you give the new or revised SOP to Quality Assurance for approval.

Procedure area, title, and SOP number:	Effective Date:
GC VOA SOP for analysis of purgeable hulographed volatile organic compounds procedure prepared by: in water by nethod 801	Ø Date:
Revised by Kelly Wans	3/17/92
Procedure approved by: (If the lab manager prepared the SOP, a	Date:
e qualified second party should sign)	· · · · · · · · · · · · · · · · · · ·
This procedure meets the requirements set forth in the following Reference mothers for evaluating solid made Surphio, 3 September 1986 (3) U.S. EPA Federal Regionstablishing test procedures for the analysis of	d edition, nevise
This procedure meets the requirements set forth in the following References motheds for evaluating solid weeks sur-1946, 3 September 1986 (2) U.S. EDA Federal Regisestablishing test procedures for the analysis of the clean Water Act. Vol. 449 part 209	ences for Approved Metr dedition, revise for Guidelines pollutents and
This procedure meets the requirements set forth in the following Reference mothers for evaluating solid made Surphio, 3 September 1986 (3) U.S. EPA Federal Regionstablishing test procedures for the analysis of	ences for Approved Metr
This procedure meets the requirements set forth in the following References motheds for evaluating solid weeks sur-1946, 3 September 1986 (2) U.S. EDA Federal Regisestablishing test procedures for the analysis of the clean Water Act. Vol. 449 part 209	ences for Approved Metr dedition, revise for Guidelines pollutents and
This procedure meets the requirements set forth in the following Reference meets the requirements set forth in the following Reference methods for evaluating solid waste Sur-BHG, 3 September 1986 (2) (1.5. EPA Federal Region establishing test procedures for the analysis of the Clean Water Act. Vol. 49 part 209 Procedure approved by Quality Assurance Representative:	ences for Approved Method Review College Colle

Section No. 4.1.32 Revision No. 1 Date: March 16, 1992 Page 1 of 14

Instrument Procedure 153, 154, 157, 165:

Analysis of Purgeable Halogenated Volatile Organic Compounds in Water by Method 8010

1.0 Scope and Application

Method 8010 provides gas chromatographic conditions for the detection of halogenated volatile organic compounds. Purgeable halogenated compounds are extracted from water by a room temperature purge and trap extraction. The trap is desorbed onto a 105-m, 0.53 mm ID capillary column, and analytes are detected by an Electrolytic Conductivity Detector (ELCD) which is mounted on a gas chromatograph. The gas chromatograph is temperature programmed, complete with a cryogenics (liquid nitrogen) system, to separate the target compounds. A surrogate is used to measure the sample recovery. Target compounds are quantitated with a multipoint and external standard calculation.

This procedure meets the requirements set forth in the following References for Approved Methods: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods; SW-846, 3rd edition, revised September 1986; and U.S. EPA Federal Register Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Final Rule, Interim Final Rule, and Proposed Rule. Vol.49, Part 209.

2.0 Target Compound List

Analyte	CAS Number
chloromethane	74-87-3
vinyl chloride	75-01-4
bromomethane	74-83-9
chloroethane	75-00-3
1,1-dichloroethene	75-35-4
methylene chloride	75-09-2
trans-1,2-dichloroethene	156-60-5
1,1-dichloroethane	75-34-3
chloroform	67-66-3
bromochloromethane	74-97-5
1,1,1-trichloroethane	71-55-6
carbon tetrachloride	56-23-5
1,2-dichloroethane	107-06-2
trichloroethene	79-01-6
1,2-dichloropropane	78-87-5
bromodichloromethane	75-27-4
dibromomethane	74-95-3
2-chloroethyl vinyl ether	110-75-8
cis-1,3-dichloropropene	10061-01-5
trans-1,3-dichloropropene	10061-02-6

Revision No. 1.32 Revision No. 1 Date: March 16, 1992 Page 2 of 14

Analyte	CAS Number
1,1,2-trichloroethane	79-00-5
tetrachloroethene	127-18-4
dibromochloromethane	124-48-1
1,2-dibromoethane	106-93-4
chlorobenzene	106-90-7
1,1,1,2-tetrachloroethane	630-20-6
bromoform	75-25-2
1,1,2,2-tetrachloroethane	7 9- 34-5
1,2,3-trichloropropane	96-18-4
bromobenzene	108-86-1
2-chlorotoluene	95-49-8
4-chlorotoluene	106-43-4
1,3-dichlorobenzene	541-73-1
1,4-dichlorobenzene	106-46-7
1,2-dichlorobenzene	95-50-1

3.0 Interferences and Safety

3.1 Interferences

Methylene chloride (which is used as an extraction solvent in other laboratories within the facility) is occasionally detected at low levels in blanks and samples analyzed by Methods 601 and 8010. CompuChem has taken measures to minimize this contamination, including physically isolating the volatile instrumentation lab, polishing incoming air through activated carbon filters built into the air handling system, maintaining a positive air pressure within the lab, and restricting access to the lab by personnel who use volatile solvents.

CompuChem has adopted an internal policy based on guidelines established by the EPA's Contract Laboratory Program which permits up to five times the detection limit for common contaminants in method blanks. The maximum methylene chloride contamination allowed in an 8010 aqueous method blank is 5 μ g/l.

Contamination can also occur by carryover whenever highlevel and low-level samples are sequentially analyzed. To prevent carryover, analyze one or more instrument blank to check and clean the systems. In extreme situations, the purge and trap system may need to be dismantled and cleaned.

Section No. 4.1.32 Revision No. 1 Date: March 16, 1992 Page 3 of 14

3.2 Safety

According to the Federal Register, the following compounds have been tentatively classified as known or suspected carcinogens:

- carbon tetrachloride
- chloroform
- = 1,4-dichlorobenzene
- vinyl chloride

Wear a gas respirator when handling samples with quantities of these compounds.

4.0 <u>Sample Preparation Screen</u>

All samples of unknown composition should be screened prior to analysis. Screening methods involve a 50:1 dilution followed by purge and trap analysis.

5.0 Procedure

5.1 Introduction of Sample

Put 5 ml of sample into a 5- or 10-ml gastight syringe with Luerlok tip (Hamilton 1005 TEFLL or equivalent). Add 5 μ ls of surrogate solution 418 with a 10- μ l gastight syringe (Hamilton 1701N or equivalent). If the screen of the sample indicates that a dilution is required, add the following sample volumes to pure laboratory water spiked with surrogate.

Dilution	Vol. of sample(ul)	Vol. of Blank Water(ml)
2:1	2500	2.5
5:1	1000	4.0
10:1	500	4.5
20:1	250	4.8
50:1	100	4.9
100:1	50	5.0
200:1	25	5.0
250:1	20	5.0
500:1	10	5.0
1000:1	5	5.0

Section No. 4.1.32 Revision No. 1 Date: March 16, 1992 Page 4 of 14

For dilutions greater than 1000:1, a serial dilution should be made. For example, a 10,000:1 dilution can be made by making a 100:1 dilution of a 100:1 dilution. The first dilution should be 100:1 or greater to minimize the risk of contaminating the syringes.

5.2 Sample Purge

The sample is injected into a purging vessel conforming to the specifications listed in the standardized methods. The sample is purged with helium at a flow of 40 ml/min for 12 minutes.

5.3 Sample Trap

The purge gas is swept through a trap containing 1.0 cm of OV-101, 7.7 cm of Tenax, and 15 cm of silica gel. An equivalent trap may be used if the trap adsorbs all the target compounds. The trap should be below 25°C during the purge procedure. The transfer line between the purge vessel and the trap should be kept above 80°C at all times.

5.4 Sample Desorption

During the desorb procedure, the target compounds are backflushed off the trap and then enter the carrier gas stream. The gas flow and trap temperature are controlled so that all the compounds are removed from the trap in as short a time as possible. When the GC is ready to start a run, the trap is preheated to 175°C then desorbed at 180°C. The backflush gas is helium flowing at 15 ml/min. The trap is desorbed for 4 minutes. The trap is then baked at 225°C for 18 minutes before being cooled to room temperature to remove water adsorbed on the silica gel during the heated purge.

Other desorb temperatures may be required with different trap material.

5.5 Chromatography

The following apparatus and conditions are used to chromatograph the target compounds.

5.5.1 GC: Varian 3400 or equivalent

5.5.2 Injection Port: Varian 1/4-inch packed column port at 200° C with Restek Uniliner and a Restek 0.80 OD insert set in the direct injection mode.

Section No. 4.1.32

Revision No. 1

Date: March 16, 1992

Page 5 of 14

5.5.3 Primary Column: Rtx-502.2 105 meter, 0.53-mm

ID, $3.00-\mu m$ film (Restek

#10910) or equivalent with the following temperature program:

(All temperatures are in

degrees C.) 0° for 4 min. Ramp to 90° at 3°/min., hold 2 min. Ramp to 130° at 2/min., hold 0 min. Ramp to 220° at 12°/min, hold 2 min. Total run time =

65.50 minutes.

5.5.4 Confirmation

Column:

Rtx-1 105 meter, 0.53 mm ID, 3.0 μ m film (Restek # 10189). Temperature program is the

same as primary column.

The temperature program above is used to separate all 8010 compounds.

5.6 Detector Parameters

The O.I. Co. model 4420 Electrolytic Conductivity Detector is used for sample detection using the following parameters:

Mode: Halogen
Make-up Gas: Helium
Reaction gas: Hydrogen
Reactor Temp: 850° C

Filter: M
Range: 1 Volt

Attenuation: 1

Detector Solvent: 1-propanol

5.7 Initial Calibration and Quantitation

Response factor (RF) = area/std concentration Sample Concentration = (area/mean RF) x dilution

Samples are quantitated using a five-level multipoint calibration with external standard quantitation. The following table lists the standard levels used to generate the multipoint response factors. The letters to the left of the compounds refer to the standards (table on the bottom of page 6) in which they are found.

<u>Compound</u>		<u>Concentrations (ug/L)</u>				
		VL	L	M1	M2	Н
A	chloromethane	4.0	8.0	16.0	24.0	32.0
A	vinyl chloride	4.0	8.0	16.0	24.0	32.0
A	bromomethane	4.0	8.0	16.0	24.0	32.0
A	chloroethane	4.0	8.0	16.0	24.0	32.0

Section No. 4.1.32

Revision No. 1

Date: March 16, 1992

Page 6 of 14

	Compound		ntrat	ions	(ug/L)	
		VL	L	M1	M2	Н
С	1,1-dichloroethene	2.0	4.0	8.0	12.0	16.0
С	methylene chloride	2.0	4.0	8.0	12.0	16.0
C	t-1,2-dichloroethene	2.0	4.0	8.0	12.0	16.0
С	1,1-dichloroethane	2.0	4.0	8.0	12.0	16.0
С	chloroform	2.0	4.0	8.0	12.0	16.0
D	bromochloromethane	2.0	4.0	8.0	12.0	16.0
С	1,1,1-trichloroethane	2.0	4.0	8.0	12.0	16.0
С	carbon tetrachloride	2.0	4.0	8.0	12.0	16.0
С	1,2-dichloroethane	2.0	4.0	8.0	12.0	16.0
C	trichloroethene	2.0	4.0	8.0	12.0	16.0
С	1,2-dichloropropane	2.0	4.0	8.0	12.0	16.0
C	bromodichloromethane	2.0	4.0	8.0	12.0	16.0
D	dibromomethane	2.0	4.0	8.0	12.0	16.0
С	2-chloroethyl vinyl ether	2.0	4.0	8.0	12.0	16.0
С	c-1,3-dichloropropene	2.0	4.0	8.0	12.0	16.0
С	t-1,3-dichloropropene	2.0	4.0	8.0	12.0	
С	1,1,2-trichloroethane	2.0	4.0	8.0	12.0	16.0
С	tetrachloroethene	2.0	4.0	8.0	12.0	16.0
С	dibromochloromethane	2.0	4.0	8.0	12.0	16.0
D	1,2-dibromoethane	2.0	4.0	8.0	12.0	16.0
C	chlorobenzene	2.0	4.0	8.0	12.0	16.0
D	1,1,1,2-tetrachloroethane	2.0	4.0	8.0	12.0	16.0
C	bromoform	2.0	4.0	8.0	12.0	16.0
С	1,1,2,2-tetrachloroethane	2.0	4.0	8.0	12.0	16.0
D	1,2,3-trichloropropane	2.0	4.0	8.0	12.0	16.0
D	bromobenzene	2.0	4.0	8.0	12.0	16.0
D	2-chlorotoluene	2.0	4.0	8.0	12.0	16.0
D	4-chlorotoluene	2.0	4.0	8.0	12.0	16.0
В	1,3-dichlorobenzene	2.0	4.0	8.0	12.0	16.0
В	1,4-dichlorobenzene	2.0	4.0	.8.0	12.0	16.0
В	1,2-dichlorobenzene	2.0	4.0	8.0	12.0	16.0

Initial calibration standards are prepared from NIST traceable concentrates according to the following table.

Microliters (μ ls) Added to 5 ml Water

	Standard	VL	L	M1	M2	Н
A	1332	1.0	2.0	4.0	6.0	8.0
В	1334	1.0	2.0	4.0	6.0	8.0
С	1328 & 30	1.0	2.0	4.0	6.0	8.0
D	8010 addtl	1.0	2.0	4.0	6.0	8.0
	418 (surrogate)	5.0	5.0	5.0	5.0	5.0

Section No. 4.1.32 Revision No. 1 Date: March 16, 1992 Page 7 of 14

If the %RSDs over the five levels are less than 20%, then the mean response factor can be used to calculate the compound concentrations (see method 8000, section 7.4.2.2). Otherwise a calibration curve must be used to calculate the sample concentrations. It is expected that all compounds in method 8010 will have an RSD of <20%. If the RSD is >20%, the following items should be checked.

- If response factors have random variation, check for leaks in the purge vessels.
- If response factors increase with concentration, check for active sites in the chromatography system.
 - If response factors from direct injections increase with concentration the active sites are probably in the GC column or injector port.
 - If response factors from direct injections remain consistent the active sites are probably in the purge and trap apparatus.
- If response factors decrease with concentration, check for contamination of the system by analyzing a blank.

5.8 Sample Identification

If single column analysis has been requested, a peak is identified as a target compound if its retention time is within the retention time window of a target compound. If a peak falls within multiple retention time windows, it is identified as the target compound with the closest relative retention time as compared to the surrogate.

If dual column analysis is requested, the sample is run on a dissimilar column if any peak is in a target compound retention time window during the first analysis.

The GC lab follows a policy that the identity of the peak is confirmed if the peak is in the retention time window for the same compound on both columns and the concentration of the compound on the second column is within - 50 to +100% of the concentration on the first column.

Exceptions to this rule can be made if there is reason to believe that matrix interference is affecting the concentration calculation on one or both columns. If an exception is made, the justification is documented, signed, and dated by the chemist performing the analysis in the form of a Laboratory Notice.

Section No. 4.1.32 Revision No. 1 Date: March 16, 1992 Page 8 of 14

5.9 Setting Retention Time Windows

Setting accurate retention time windows is one of the most important procedures for the identification of target analytes. The procedure is covered by section 7.5 in method 8000 and paragraph 10.10 in method 601. Both methods use three times the standard deviation of three injections of a check standard over a 72-hour period. Method 601 says this procedure "can be used to calculate a suggested window size." Method 8000 says this procedure "will be used to define the retention time window." Both methods say the experience of the analyst should weigh heavily in the interpretation of chromatograms.

The procedure specified in the methods, determined by analyzing replicate check standards, will set a window that is too restrictive, increasing the risk of false negatives.

This is because analysis of check standards does not incorporate all the variables that can affect retention time.

Variables affecting retention times can be divided into two classes: system dependent and matrix dependent.

5.9.1 System-dependent variables

System-dependent variables can be monitored and, to a certain extent, controlled by the GC chemist. Recent advances in GC technology have enhanced the ability of the chemist to control many system variables. The following system attributes have been found to affect retention times.

- Carrier gas flow rate: A consistent flow rate is important. The flow rate can be affected by changes in line pressure, leaks in the system, or changes in backpressure.
- Carrier gas density: Carrier gas density can have a surprising effect on retention time. In general, increasing the flow, molecular weight, and isothermal hold times will increase the effects of changes in density. The critical point where density is variable is at the flow controllers which are usually at "room" temperature. Therefore, precise control over the temperature of the lab can be important to retention time stability.

Section No. 4.1.32 Revision No. 1 Date: March 16, 1992 Page 9 of 14

- Oven temperature: Oven temperature is the most important parameter affecting retention times. As such, it is an area where GC manufacturers have improved by producing very stable and uniform oven temperatures and heating ramps. In the volatile analysis, the Varian 3400 can achieve a stable isothermal temperature down to 35°C. Below that point, liquid nitrogen is required as a coolant. is also critical that the oven temperature stabilizes for 2 minutes before the sample is desorbed. Therefore, the purge and trap should advance to "desorb preheat" after receiving a "GC ready" signal from the GC instead of relying on a thermocouple connection and minimum temperature cut-off.
- Desorb time: Controllable changes in the desorb time are caused by changes in the trap temperature and desorb gas flow. The desorb gas is controlled by the GC and is affected by all the parameters that affect carrier gas. The trap temperature is held at ambient levels during purge and ballistically heated during desorb preheat and desorb. Therefore, it is important that the ambient temperature remains stable. The heating profile of the trap is not important during desorb preheat because the target compounds are not moving during this period; however, it is important during desorb. Therefore, the preheat temperature must be close to the final desorb temperature.

5.9.2 Matrix-dependent variables

The sample matrix affects the desorb time and the column characteristics. Polar compounds in a matrix can coat the surface of the capillary liquid phase so that the target compounds skip along the column rather than plow through the phase.

Because of all these factors, each GC system has unique retention time windows. The window should be set at ± 1.5% of the retention time of each compound from each continuing calibration check standard.

Section No. 4.1.32 Revision No. 1 Date: March 16, 1992 Page 10 of 14

6.0 Quality Control

6.1 Surrogates (ID # 418)

The surrogates for method 8010 differ from method 601 because Trichlorofluoromethane (TCFM), a current surrogate for 601, is on the compound list for 8010. Because CompuChem does not validate this compound at this time (we do not see TCFM in water), TCFM and Bromofluorobenzene are used as the surrogates. TCFM is spiked at 10.0 μ g/L, and historically generated 601 data indicate that the acceptance range is 76%-135%. Bromofluorobenzene is spiked at 30.0 μ g/L and the acceptance range is 69%-123%. This range will be periodically updated as historical method performance data are accumulated.

6.2 Continuing Calibration Check Standards

In accordance with method 8010 requirements, a continuing calibration check standard is run after every 10 production samples. The continuing calibration check standards are prepared at the M1 concentration level found in section 5.7. The check standards contain all compounds listed in section 2.0 of this SOP.

The acceptance criteria is taken from Table 3 of method 8010. Historically generated laboratory acceptance criteria will be used for any additional compounds. The table below compares method 8010 acceptance criteria and historically generated laboratory acceptance criteria.

Calibration Acceptance Criteria For Check Standards

	8010 %	Lab 🕏
Compound	recovery range	recovery range
chloromethane	D-193	1-193
vinyl chloride	28-163	31-170
bromomethane	D-144	1-180
chloroethane	38-150	39-139
1,1-dichloroethene	28-167	25-170
methylene chloride	25-162	25-165
t-1,2-dichloroethene	38-155	40-154
1,1-dichloroethane	47-132	50-130
chloroform	49-133	50-130
bromochloromethane	47-132 ^a	47-132
1,1,1-trichloroethane	41-138	45-135
carbon tetrachloride	43-143	40-141
1,2-dichloroethane	51-147	50-150
trichloroethene	35-146	35-145
1,2-dichloropropane	44-156	46-160
bromodichloromethane	42-172	42-170

Section No. 4.1.32 Revision No. 1 Date: March 16, 1992

Page 11 of 14

Compound	8010 % recovery range	Lab % recovery range
dibromomethane	42-172 ^a	42-172
2-chloroethyl vinyl ether c-1,3-dichloropropene	14-186 22-178	15-200 20-180
t-1,3-dichloropropene 1,1,2-trichloroethane	22-178 39-136	20-185 40-135
tetrachloroethene dibromochloromethane	26-162	20-170 20-190
1,2-dibromoethane	24-191 24-191 ^a	24-191
chlorobenzene 1,1,1,2-tetrachloroethane	38-150 8-184 ^a	35-150 8-18 4
bromoform 1,1,2,2-tetrachloroethane	13-159 8-184	10-170 5-190
1,2,3-trichloropropane	60-140 ^a	60-140
bromobenzene 2-chlorotoluene	60-140 ^a 60-140 ^a	60-140 60-140
4-chlorotoluene 1,3-dichlorobenzene	60-140 ^a 7-187	60-140 5-190
1,4-dichlorobenzene 1,2-dichlorobenzene	42-143 D-208b	45-140 1-200

a Value taken from CompuChem's historically generated acceptance range.

When a check standard fails to meet the recovery criteria, an initial calibration must be performed. Failure of the check standard means analysis cannot continue until system maintenance is performed and the check standard passes criteria.

6.3 Blanks

Method 8000 specifies that one method blank be prepared per analytical batch (up to 20 samples). For method 8010, a blank will be run either once per day per instrument or once per analytical batch, whichever is more frequent. A method blank consists of laboratory pure water spiked with bromofluorobenzene (BFB) and trichlorofluoromethane (TCFM) at 5 μ l per 5.0 ml of water. BFB and TCFM % recovery must fall within the acceptance ranges found in section 6.1. The method blank must not contain any target compounds above the reporting limit. Exceptions to this are common laboratory solvents (i.e., methylene chloride) which may be present in blanks at 5X the stated detection limit.

 $^{^{}b}$ D = Detected.

Section No. 4.1.32 Revision No. 1 Date: March 16, 1992 Page 12 of 14

6.4 Matrix Spikes

As specified in method 8000 (paragraph 8.5), a matrix spike and matrix spike duplicate will be analyzed per analytical batch (up to 20 samples). The MS/MSD is prepared by separately spiking two 5-ml aliquots of sample with all target analytes at the M1 concentration level. The second table in section 5.7 specifies how much of each standard concentrate to add. The MS/MSD must pass the acceptance criteria listed in section 6.2 for a continuing calibration standard. If the MS/MSD fails acceptance criteria a blank spike should be performed to verify that the GC system is in control. If the blank spike does not meet acceptance criteria, analysis cannot be continued until after system maintenance has been performed and a blank spike passes criteria. Then the duplicate matrix spikes should be reanalyzed. All samples associated with a failed blank spike must also be re-analyzed. Blank spikes must contain the same compounds at the same levels as the check standard.

7.0 <u>Calculations</u>

Reponse factor (RF) = area of compound to be measured conc. of the compound to be measured in the standard

Method 8010 uses two internal standards, trichlorofluoromethane (TCFM) and bromofluorobenzene (BFB).

TCFM correction factor(CF) = area of TCFM in sample

(avg. RF of TCFM from multipoint/10)

BFB correction factor(CF) = area of BFB in sample

(avg. RF of BFB from multipoint/10)

Compound quantitation equation =

area of compound in sample
avg. RF of compound in sample x TCFM CF

area of compound in sample
avg. RF of compound in sample x BFB CF

Revision No. 1.32 Revision No. 1 Date: March 16, 1992 Page 13 of 14

Use equation (2) for the quantitation of 1,2-dichlorobenzene, 1,3-dichlorobenzene, and 1,4-dichlorobenzene. All other 8010 compounds are quantitated using equation (1).

TCFM % recovery calculation: TCFM CF

---- x 100

BFB CF

BFB % recovery calculation: BFB CF

--- x 100

TCFM CF

All quantitations are rounded to two significant figures.

8.0 <u>Method Validation Summary</u>

The following table summarizes the results of the method validation study performed June 5-6, 1989. The reporting level has been set by the validation data. The study was performed following the EPA requirements set forth in the Federal Register (Vol. 49, No. 209, Appendix B to Part 136, pp.43430-43431), and CompuChem's QA Program Plan, Appendix A.

Compound	Average %	MDL	Report
	Recovery	(µg/l)	(ug/l)
chloromethane vinyl chloride bromomethane chloroethane 1,1-dichloroethene	69.8 69.8 78.3 85.3	0.495887 0.541896 0.440651 0.495887 0.303800	0.50 0.55 0.45 0.50 0.35
methylene chloride t-1,2-dichloroethene 1,1-dichloroethane	102.0 85.5 93.0	0.303800 0.797334 0.294521 0.338500	1.0 0.30
chloroform bromochloromethane 1,1,1-trichloroethane	84.5	0.320404	0.35
	78.5	0.220327	0.25
	86.0	0.303831	0.35
carbon tetrachloride 1,2-dichloroethane trichloroethene 1,2-dichloropropane	83.5	0.310310	0.35
	79.0	0.211100	0.25
	87.0	0.282100	0.30
	76.0	0.276400	0.30
bromodichloromethane	80.0	0.398949	0.40
dibromomethane	87.5	0.383698	0.40
2-chloroethyl vinyl ether	88.5	0.379526	0.40
c-1,3-dichloropropene	60.0	0.252318	0.30
t-1,3-dichloropropene	81.5	0.234330	0.25
1,1,2-trichloroethane	85.0	0.218514	0.25
tetrachloroethene	89.0	0.276400	0.30
dibromochloromethane 1,2-dibromoethane chlorobenzene	63.0	0.258549	0.30
	84.0	0.328980	0.35
	84.0	0.303830	0.35

Section No. 4.1.32 Revision No. 1 Date: March 16, 1992 Page 14 of 14

	Average &	MDL	Report
Compound	Recovery	<u>(µg/1)</u>	(Ha/1)
1,1,1,2-tetrachloroethane	85.5	0.344348	0.35
bromoform	88.5	0.478210	0.50
1,1,2,2-tetrachloroethane	88.5	0.399944	0.40
1,2,3-trichloropropane	93.5	0.310310	0.35
bromobenzene	71.4	0.817600	0.85
2-chlorotoluene	74.0	0.211104	0.25
4-chlorotoluene	89.0	0.303830	0.35
1,3-dichlorobenzene	109.0	0.182025	0.20
1,4-dichlorobenzene	112.0	0.198360	0.20
1,2-dichlorobenzene	111.0	0.265346	0.30

Section No. 5.0 Revision No. 5 Date: June 20, 1991 Page 38 of 56

Attachment #7

QC SPIKE RECOVERY CONTROL LINITS

AND NATRIX SPIKE DUPLICATE RELATIVE PERCENT DIFFERENCE (RPD) LINITS

METHOD 8010, VOLATILE HALOCARBONS

(40 CFR 136, Oct. 26, 1984 Federal Register)

Compound	Aqueous (\$ Recovery)	Aqueous (RPD)	Solid (% Recovery)	Solid (RPD)
Bromodichloromethane	42-172	20	42-172	20
Bromobenzene	60-140	20	60-140	20
Bronoform	13-159	20	13-159	30
Bromomethane	D-144	20	D-144	20
Carbon Tetrachloride	43-143	20	43-143	20
Chlorobenzene	38-150	20	38-150	30
Chloroethane	46-137	20	46-137	20
2-Chloroethylvinyl Ether	14-186	20	14-186	20
Chloroform	49-153	20	41-153	20
Chloromethane	D-193	20	D-193	20
2-Chlorotoluene	60-140	20	60-140	20
4-Chlorotoluene	60-140	20	60-140	20
Dibromochloromethane	24-191	20	24-191	20
1,2-Dibromoethane	24-191	20	24-191	20
1,2-Dichlorobensene	D-208	20	D-208	20
1,3-Dichlorobensene	7-147	20	7-187	20
1,4-Dichlorobenzene	43-143	20	42-143	20
1,4-Dichlorobutane	80-120	20	80-120	20
1,1-Dishloroethene	28-167	20	28-167	20
1,2-Dichloroethane	51-147	20	51-147	20
1,1-Dichlorosthane	47-132	20	47-132	20
trans-1,2-Dichloroethene	38-155	20	38-155	20
1,2-Dichloropropane	44-156	20	44-156	20
cis-1,3-Dichloropropene	22-178	20	22-178	20
trans-1,3-Dichloropropene	22-178	20	22-178	20
Nethylene Chloride	25-162	20	25-162	20
1,1,3,2-Tetrachloroethane	8-184	20	8-184	20
Tetrachloroethene	26-162	20	26-162	. 20
1,1,1-Trichlorosthane	41-138	20	41-138	20
1,1,2-Trichloroethane	39-136	20	39-136	20
Trichloroethene	35-146	20	38-146	20
richlorofluoromethane	21-156	20	21-156	20
1,2,3-Trichloropropane	60-140	20	60-140	20
Vinyl Chloride	26-163	20	26-163	20

DELIVERABLES CODE FOR REPORT FORMAT

Style 9 includes:

- o Cover letter
- o Table of Contents
- o Chronicle (including QC summary)
- o Case Narrative
- o Method Reference
- o QA Notices (if applicable)
- o Chain-of-Custody (if received)
- o Compound List
- o RIC (sample)
- o Quant. Report
- o Spectra
- o Library Search Form IV (if applicable)
- o Library Scarch Spectra (if applicable)
- o Blank Compound List
- o RIC (blank)
- o Quant. Report
- o Spectra
- o Matrix Spike/Matrix Spike Duplicate
- o RIC (spikes)
- o Quant. Report
- o Tuning Summary (for sample, blank & spikes)
- o Calibrations (Initial & Continuing for sample blank & spikes)
- o RIC (standards: includes, in order, samples, blank & spike)
- O Quant. Report (sample, blank & spike)

APPENDIX A.3

COMPUCHEM LABORATORIES, INC.

CHAIN-OF-CUSTODY PROCEDURES

Section: Appendix E

Revision No. 2

Date: October 3, 1988

Page 1 of 2

Chain-of-Custody

語· | 新子 | 獨一一樣等 | 紹子 | 傳達 | 稀子 | 梅香 | 野

1 7

÷

The basic components for maintaining sample chain-of-custody (C-O-C) are:

- 1) samples must be relinquished into the possession of an authorized laboratory staff member, or
- 2) samples must be within the authorized staff member's line-of-sight, or
- 3) samples must be locked in a secured storage area with restricted access.

Furthermore, any change of possession or custody must be documented on appropriate chain-of-custody forms. This documentation must include both the initials of the individual relinquishing the sample and those of the individual receiving the sample, as well as the date of the custody transfer.

CompuChem® accomplishes these objectives through an elaborate document control system. This system includes procedures for documentation of the receipt of the sample into the laboratory using preprinted, numbered chain-of-custody records (although many clients provide their own C-O-C records which suffice). These records include information about the individuals collecting the samples, the collection date, time and location, and the type of analyses required. CompuChem's clients are responsible for field chain-of-custody, sample collection, handling and shipping.

When the samples are received in the laboratory, the C-O-C documents are signed and dated by the Receiving Clerk. The samples are logged into the Computerized Laboratory Management System (CLMS), and assigned unique sample identification numbers. The samples are then relinquished to the possession of a Sample Custodian, who has sole access to the locked sample storage refrigerators. The CLMS schedules the appropriate analyses and tracks the progress of sample

Section: Appendix E Revision No. 2

Date: October 3, 1988

Page 2 of 2

processing throughout the laboratory. Samples do not remain outside refrigeration more than 2 hours from receipt.

•

11.

. .

;~

1.7

٣,

_;

\$ 12

* :

3 3

.

The custody of each sample can be determined at any point in time by reviewing Scheduling Details within the CLMS. A "paper trail" also accompanies the movement of the sample (or extracts, aliquots or digestates created from the as-received sample) throughout the lab, serving to document internally all changes in custody.

The integrity of the samples within the laboratory is assured by the security of the facility itself. Building security is controlled by an electronic card entry system. The exterior doors and the doors of various controlled-access areas are equipped with card readers. Each member of the staff has an access—card, which must be prominently displayed on their person, that is coded only for those areas where their job functions require access. The system also maintains a record of the movements, or attempted movements, of the staff throughout the building. A computer printout of this record is audited by a member of the Quality Assurance Department for verification of card coding and card entry transactions.

When the analysis is complete, the final extracts (for extractable portions of the sample) are kept in a locked freezer (if required) under sole custody of the Sample Custodian.

A complete description of CompuChem's sample tracking procedures and additional chain-of-custody details can be found in the Production, Planning and Control SOPs.

APPENDIX A.4

COMPUCHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURES FOR PRODUCTION PLANNING AND CONTROL

Section No. 1.2 Revision No. 3 Date: May 1, 1989 Page 1 of 2

Production Planning & Control SOP 1.2: Storing Samples

The walk-in refrigerator at CompuChem Laboatories is kept at 4 (+2) degrees centigrade to ensure sample stability. The temperature of the walk-in cooler is recorded daily in the "Daily Walk-In Cooler Log" by the Scheduling Clerk/Custodian and reviewed monthly by the Scheduling Supervisor. The Scheduling Clerk/Custodian enters the date, temperature, and signs the entry. In the event that the temperature is outside the acceptable range (2 to 6 degrees centigrade):

- 1. Check refrigerator fan operation. If not operating, call Facility Personnel.
- 2. Close doors and check temperature in ½ hour. If the temperature is still not within acceptable range, call Facility Personnel. Facility Personnel: 596-3729 or 596-1917.

The walk-in is locked at all times and only appropriate Shipping and Receiving and Production Planning and Control personnel are issued keys. The Sample Custodian arranges samples in the walk-in first by Receipt date and then according to container size. There are two separate refrigerators for raw sample storage. Cooler #1 contains all extractable and inorganic samples. These are stored according to sample receipt date and container size. All samples requiring volatile analysis are stored in a separate refrigerator Cooler #2. These samples are also stored by sample receipt date. All volatile water samples are inverted for storage.

The sample stock is rotated daily by due date to aid in the timely processing of all samples.

Shipping and Receiving is responsible for monitoring the effectiveness of the activated carbon filter in the walk-in. To detect the possible presence of volatile contaminants in the walk-in, 20 sample bottles (each filled with

Section No. 1.2 Revision No. 3 Date: May 1, 1989 Page 2 of 2

sparged, deionized water) are put into the walk-in 24 hours after the carbon filter has been changed. One bottle per week is removed from the walk-in and analyzed by the GC/MS lab for volatiles. If for three consecutive weeks these analyses detect contamination greater than the detection limit for a compound (particularly methylene chloride), the Manager of Quality Assurance is notified and the filter is changed. After the filter is changed, this monitoring process is repeated.

Section No. 1.5 Revision No. 2 Date: May 30, 1989 Page 1 of 21

Production Planning and Control SOP 1.5: SampleSaver® Preparation

A SampleSaver® is sent to most commercial customers requesting that CompuChem analyze a group of samples. These orders are taken by a Customer Service Representative, who enters the orders into the CLMS system. The system then generates a worklist for each order. The SampleSaver® Worklist contains the following information (refer to Attachments 1-2):

* Address of the client

- * Type of SampleSaver® to be sent (see list below)
- Special instructions: use of chain-of-custody, etc.
- * Method of shipment
- * Account number
- * Latest shipping date
- * Analysis codes for samples
- * SampleSaver® number: this is assigned by the CLMS and appears on the order received from the system.

SampleSaver® Number is hand-written on an adhesive label which is attached by receiving personnel to the sample container(s). An information packet is included, and consists of the Client Information Sheet, instructions for using SampleSaver® materials (these vary according to the type of SampleSaver® that is sent), Sample Collection Procedures (sent with all types of SampleSavers®), a Chain-of-Custody Record, Chain-of-Custody Seals, Sample I.D. labels, return address labels, and hazardous shipping labels (see Attachments 3-12).

SampleSaver[®] configurations required by clients may include a preservative kit (see Production Planning and Control SOP 3.6), laboratory pure water or Ottawa sand blanks (see Production Planning and Control SOP 3.7).

Section No. 1.5 Revision No. 2 Date: May 30, 1989 Page 2 of 21

If the client requests that the Chain-of-Custody Record originate from CompuChem, the Supervisor of Sample Receiving signs and dates the Record (in the "Relinquished By" box), which initiates the chain-of-custody process. The SampleSaver is sealed with chain-of-custody tape. When the SampleSaver® is returned, PP&C SOP #1.1 is followed to continue this process.

The configuration of a SampleSaver® is dependent on the SampleSaver® Code on the worklist. The following codes are a list of the SampleSaver®'s possible configurations:

Section No. 1.5 Revision No. 2 Date: May 30, 1989 Page 3 of 21

SAMPLESAVER® CONFIGURATIONS

001 THRU 009 SINGLE ITEM TYPE SAMPLESAVERS 010 THRU 019 WATERS WITH CLEAR LITER BOTTLES

010 111110		W	III QEEMI E	11511 0	V L L	. .		
SAMPLESAVER CODE	LI CLEAR	TERS AMBER	500 ml PLASTICS	4 oz Jar	VOA	40 ml VIA CYANIDE	LS PHENOL	TRIP BLANK
000	DU	MMY SAMPL	E SAVER					
001	4							
002		4						
003			6					
004				10		<u> </u>		
005					8			
006	1				16			
007					24	1		
008		1			32		1	
009		1		1	40			
010	4	1	2.	1		1		
011	4	1		1		l		
012	4	1						
013	4	1	2	1		l		
014	4		2					
015	4		2			1		1
016	4		2					1
* 017	1				1			
* 018								
* 019								1

^{*} Codes for configurations yet to be standardized.

Section No. 1.5 Revision No. 2 Date: May 30, 1989 Page 4 of 21

SAMPLESAVER® CONFIGURATIONS

020 THRU 029 WATERS WITH AMBER LITER BOTTLES 030 THRU 039 SOILS

U3U INKU	033	20162						
SAMPLESAVER CODE	LI CLEAR	TERS AMBER	500 ml PLASTICS	4 oz Jar	VOA	40 ml VI CYANIDE	ALS PHENOL	TRIP BLANK
020		4	2					***
021		4			8			
022		4			7			1
023		4	2		5	1	1	1
024		4		2				
025		4	2	J	8			
026	1	4	2	1	7			1
* 027	1							
* 028								
* 029								
030	4		2					
031	4			1				
032	4			2				
033	3		1	4				
034	2			6				
035	1		1	8				
* 036			1					
* 037								
* 038								
* 039					1			

^{*} Codes for configurations yet to be standardized.

Section No. 1.5 Revision No. 2 Date: May 30, 1989 Page 5 of 21

SAMPLESAVER® CONFIGURATIONS

LITERS - DISTILLED VOA - SPARGED

040 THRU 049 WATER FILLED BOTTLES (WATER BLANKS) PLASTICS - DEIONIZED 050 THRU 056 FURNACED DIRT FILLED BOTTLES (SOIL BLANKS)

SAMPLESAVER CODE	LI	TERS AMBER	500 ml PLASTICS	4 oz Jar	VOA	40 ml VI CYANIDE	ALS PHENOL	TRIP BLANK	. — —
040	4	1							•
041	4		2						•
042	4			1	8				_
043	4		2	1	7			1	_
044	l	4							
045		4	2	l		·			
046		j 4	1		8		1		_
047	ĺ	4	2	l	7		l	1	•
* 048			1	1					•
* 049				1					•
* 050	4		1					1	•
051	4	1	2	1					
052	4		1.	2					_
053	3			4		1			
054	2			6					
055	1			8					
056				10					
099		SPECIAL (ONF I GURAT	ION					-
100		BULK SHIF	PMENT OF GL	.ASSWAF	RE/PLA	STICS			
201	(GROUND WAT	TER MONITOR	RING RE	QUIRI	ING 2 SAME	PLESAVERS	5	

 $[\]star$ Codes for configurations yet to be standardized.

Section No. 1.5 Revision No. 2 Date: May 30, 1989 Page 6 of 21

060 THRU 069 PRESERVATIVE KITS

SAMPLESAVER	l		PRE:	SERVATIVE	KITS		
CODE	HNO3	HC1	HOSM	H ₂ SO ₄	Zn(C2H3O2)2		
060	1	1	1	1			
061	1	1					
062			1				
063	1			1			
064	1		1	1			
065	1	1	1	1			
066	1	1	1				
* 067	1			·			
* 068	}						
069) AS R	EQUESTED	FOR SUB-CO	NTRACTED A	ANALYSIS		

^{*} Codes for configurations yet to be standardized.

ниоз	NITRIC ACID
нсі	HYDROCHLORIC ACID
NaOH	SODIUM HYDROXIDE
H2S04	SULFURIC ACID
Zn(C2H3O2)2	ZINC ACETATE

Section No. 1.5 Revision No. 2 Date: May 30, 1989 Page 7 of 21

SAMPLESAVER® CONFIGURATIONS

070 THRU 079 MINI-SAMPLESAVERS 080 THRU 089 MINI-SAMPLESAVERS

080 1860	089	MINI-2WML	LE2AVEK2					
SAMPLESAVER CODE	LI CLEAR	TERS	500 ml PLASTICS	4 oz Jar		40 ml VI CYANIDE		TRIP BLANK
070	1		1 .		2-8			1
071	1	1	1		2-7			
072	2				4			
073	2							
074	1	1		2				
075	1	1	1	1				
076	1	1		10				
077	1	İ	3					
078	1	1	[1	24		1	
* 079		1		1				
080	İ	2		l	4			l
081	1	2		2	2-8		1	1
082		1	1		2-7		ļ	1
083		1	1					1
084		1	2					1
085		1	1	2				
* 086								
* 087								
* 088								
* 089								
090		SPECIAL N	INI-SAMPLE	SAVER	CONF I	GURATION		,

^{*} Codes for configurations yet to be standardized.

Section No. 1.5 Revision No. 2 Date: May 30, 1989 Page 8 of 21

SAMPLESAVER® CONFIGURATIONS

90 SPECIAL MINI-SAMPLESAVER CONFIGURATION
091 THRU 096 MINI-SAMPLESAVERS FILLED W/WATER
096 THRU 099 MINI-SAMPLESAVERS FILLED W/FURNACED DIRT

SAMPLESAVER CODE	CLEAR	TERS R AMBER	500 ml PLASTICS			40 ml VI CYANIDE		TRIP BLANK	
090	SPE	CIAL CONF	IGURATION	MINI-S	AMPLE	SAVERS			•
091	1	1	1		2-7				•
092	2	1	}		4				•
093	2	1	1	1		,	1		•
094	1	2							•
095	1], 1] 1	1	2-7		l	1	•
096	2	١.			1			}	••
097	l] 2	1	1					•
098		1		2-12					•
* 099			1						_
									_

^{*} Codes for configurations yet to be standardized.

Section No. 1.5 Revision No. 2 Date: May 30, 1989 Page 9 of 21

To make up these configurations, Shipping and Receiving keeps on hand the necessary glassware for SampleSavers. Maintaining this glassware stock requires that Glassware Preparation be informed daily of Shipping and Receiving's needs.

Attachment #1
Section No. 1.5
Revision No. 2
Date: May 30, 1989
Page 10 of 21

SampleSaver® Worklist for RTP

Shipping Method: FED EXPRESS - SPECIAL (12) Priority: SA

Ship NLT

题, 图, 经分配。图, 图, 图, 图, 图, 图

Date Account:

Ship to:

Order: Status: Entered

Receiving Plant:
SampleSaver® Codes:
Sample Savers:
Employee Number

SampleSaver® SampleSaver® Return/Air Number Code Bill

Shipping/Receiving Instructions

Attachment #2 Section No. 1.5 Revision No. 2 Date: May 30, 1989 Page 11 of 21

SampleSaver® Picklist for RTP

Shipping Method: FED EXPRESS - SPECIAL (12) Priority: SA

Ship NLT

Date Account: . Ship to:

Order:

Status:

Receiving Plant:

Entered

SampleSaver[®] SampleSaver[®] Ship Air Bill Number Code

Shipping/Receiving Instructions

Attachment #3
Section No. 1.5
Revision No. 2
Date: May 30, 1989
Page 12 of 21

SAMPLE COLLECTION PROCEDURES

This sheet is provided to illustrate a typical sample collection procedure. For further details, refer to your Regional EPA Office or the following Federal Register issues: June 14, 1979; May 19, 1980.

The CompuChem SampleSaver® is packed in a variety of configurations dependent upon the analysis requested.

The transfer of the transfer o

Current EPA Regulations call for samples to be collected on a 24-hour compositing basis for all sample fractions, except for total phenol and cyanide vials. All containers in the SampleSaver® should be filled during a normal facility operational cycle with the total sampling cycle not exceeding a 24-hour time period.

After collection, each container should hold a "COMPOSITE" sample which represents a composite of the discharge flow during the 24-hour period. That is, the total volume of sample in the container is made up of parts with the volume of each part proportional to the flow of the discharge. For example, if 50% of the discharge flow occurs in the first hour of sample collection, then one half of the volume of each sample container (except the VOA, cyanide, and total phenol vials) should be filled with sample collected during that hour. If your discharge flows only eight hours a day and the flow for the final seven hours is uniform, the remaining half of the sample containers should be filled with seven equal portions of sample collected at seven one hour intervals. You should collect at least eight portions of sample to generate the composite sample.

For the VOA vials, the procedure is similar except that each portion of the VOA sample is collected in an individual vial. When the vials are received at CompuChem, EPA protocols require that we composite all vials into one sample just prior to analysis.

The VOA vials should be filled completely, once each vial is opened, during a period not exceeding fifteen (15) minutes (the sample is called a "GRAB" sample). For VOAs it is the number of vials filled that is done on a flow proportional basis. Using the same flow discharge example, if your SampleSaver® contains six (6) VOA vials, you should grab-fill three (3) vials the first hour of sampling. The remaining three (3) vials would be filled at three (3) equally spaced intervals during the next seven (7) hours. When received by our laboratory, compositing of the six (6) vials into one (1) container will result in a sample whose contents are proportional to the discharge flow.

Finally, if you are requesting cyanides and total phenols, the container for each of these samples is filled as a grab sample at a randomly selected time. The collection period should not exceed fifteen (15) minutes.

CLIENT INFORMATION SHEET



## 	•	Revision No. 2 Date: May 30, 1989 Page 13 of 21
		Attachment #4
	CLIENT INFORMATION SHEET	COMPUCHEM LABORATORIES
	•	3308 Chapel Hill/Nelson Highway P.O. Box 12652 Research Triangle Park, North Carolina 27709 Telephone 919/549-8263
At the end of the sampling persample via express transportations of the sampling persample via express transportation. Please complete this form and reference the sampling persample via express transportation.	ion.	•
ě		
To: Date		
	•	
vddress		Zip Code
Sample Name/Number*		

*(sample ID can be no longer than 10 characters in length)

IMPORTANT All preservatives to be added at the time of sampling by the client!!!

Section No. 1.5 Revision No. 2 Date: May 30, 1989 Page 14 of 21

COMPUCHEM LABORATORIES

THE SAMPLESAVER. AND HOW TO USE IT TO YOUR ADVANTAGE



Thank you for deciding to use our patented SAMPLESAVER for the shipment and collection of your environmental sample.

Use of the SAMPLESAVER provides you with two major advantages. First, it's easier to collect and ship a sample. Second, by properly using the SAMPLE-SAVER, your sample will more closely follow sample preservation protocols. The SAMPLESAVER is designed to maintain your sample at 4 c for 72 hours.

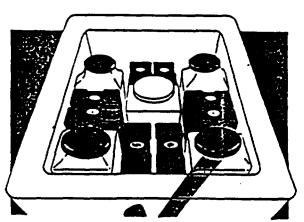
The charge for a SAMPLESAVER is only a usage charge. We retain possession of all the pieces. Usage fees cover decontamination, handling, and in some cases freight cost.

Every part of the SAMPLESAVER is crucial to the survival of your sample. Failure to return all parts could result in several things happening: sample container breakage, samples which do not meet preservation protocols and regretfully, an additional charge of \$150.00 for damage or loss of the SAMPLESAVER or its parts.

To help you get full benefit from the SAMPLESAVER, please review and follow these instructions:

THE SAMPLESAVER

 Upon receipt, open it and examine the packing arrangement. That's the way it should be repacked prior to shipment.



THE FOAM CONTAINER

- Use only the bottles we provide. Do not alter the foam container to accept any other bottles.
- Do not write on the foam container itself. Space is provided on the lid cover sheet on the enclosed forms for company and a ten character sample i.D. Any other marking may cause incorrect identification of an incoming sample.

COMPUCHEM LABORATORIES IMPORTANT! PLEASE READ ALL INFORMATION BEFORE SAMPLING. FILL OUT AND RETURN ALL ENCLOSED FORMS WITH YOUR SAMPLE COMPANY

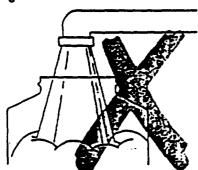
 Always keep the foam lid matched with the foam box—there are different models and the lids are not interchangeable.

BOTTLES

• Do not open any bottles until you actually put your sample in them—this prevents contamination.



- Do not substitute your own bottles or interchange any lids or labels on our bottles.
- Repack all bottles we provided, especially the plastic bottles in the center. Otherwise, bottle breakage may occur.
- Do not rinse our bottles prior to sample collection.
 Rinsing will contaminate them.



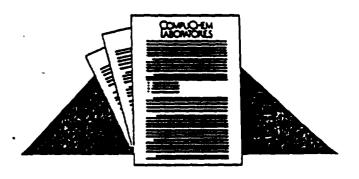
"BLUE" ICE SUBSTITUTE

- Freeze'in a standard freezer for at least 12 hours and no more than 18 hours prior to sampling. Do not freeze the blue ice using dry ice. This freezes your sample and breaks bottles.
- Do not freeze the sample itself. Our blue ice will do the job if you pack it correctly.
- Do not use regular ice or other ice substitutes.
- Please repack the blue ice (with red caps upward).
 Otherwise, your sample results will not be accurate or bottle breakage may occur during shipment.



SAMPLE RECORDS

• Return all forms properly and fully completed.



- Four types of labels will be supplied. Included will be: a return label, Chain of Custody seals, hazardous shipping labels, and a sufficient number of sample identification labels. On the same identification labels, please indicate the analysis code you have ordered and your sample identification (the sample ID can be no more than ten characters). Affix these labels to the appropriate sample containers.
- A Chain of Custody Record and Client Information Sheet are also provided. The Analysis Code ordered as well as the volume requirements will be stated on the Client Information Sheet. In order to eliminate any possible confusion about your samples, please complete each of the forms provided.
- Provide as much identifying information about your company and your sample identity as possible.
 CompuChem® processes thousands of samples a year—often from several plants within several divisions of the same company, all at the same time!

The SAMPLESAVER is unique in our business. If you have any other questions regarding the use of this SAMPLESAVER please call your customer service representative at 800-334-8525.

COMPUCHEM LABORATORIES ANALYTICAL SERVICES

3308 Chapel Hill/Nelson Highway, P. O. Box 12652, Research Triangle Park, North Carolina 27709. Telephone 919-549-8263



Attachment # 6

Section No. 1.5 Revision No. 2 Date: May 30, 1989 Page 15 of 21

PROJ. N		HOJEC						NO. OF CON-		/						Se R	ttachment #·7 ection No. 1.5 evision No. 2 ate; may 30, 1989
5TA. NO.	DATE	TIME	8	GAAS		STATIO	N LOCATION	TAINERS	/	_	_	\angle	_	_		Pa	age 16 of 21
								-						_	<u></u>		
							•										
			-				<u> </u>		 			_				<u>-</u> :	
			Ŀ			•											
			_			· · ·							-				
														_			
							•										
		-				,, ,, ,, ,,		 	<u> </u>			-					
Relinquist	red by: /	Signature	;		Date	Time	Received by: (5/presure)	Rell	nquisi	ned b	y: (S)) W) Berg	- j	Date	/Time	Received by: (Signeture)
Relinquist	red by: (Signe ture	·)	_	Dete	Time	Received by: (Signerare		Reli	tiupn	ned b	y: Isa	rie Duri	•)	Date	/Time	Received by: (Signature)
Relingulat	ned by:	Signature	·)	+	Date /	Time	Received for Laborato	ry by:		Date	• /Ti	me	u	omari	<u> </u>	l	<u> </u>
	 						Shipment; Copy to	Floid File			-1-		\dashv				

Attachment as Section 1.1.5 Revision 1.1 Date: Ma. 1.1499 Page 17 of 11



CompuChem Laboratories P.O. Box 12652 Chapel Hill/Nelson Highway Research Triangle Park, NC 27709



CUSTODY SEAL

Signature

Ď.at.

CUSTODY SEAL

Signature

Date

CUSTODY SEAL

Signature

Date

COMPUCHEM LABORATORIES

SAMPLING

___ DATE _

COMPUCHEM LABORATORIES

RETURN POSTA	GE GUARANTEED
-	•

	•		= 12 (11 (11 (11 (11 (11 (11 (11 (11 (11	
-ID#	SAMPLING DATE	S-10#	SAMPLING DATE	
MALYZE FOR/CODE	·	ANALYZE FOR/CODE:		_
D#	SAMPLING DATE	S-ID#.	SAMPLING DATE	
FALYZE FOR/CODE		ANALYZE FOR/CODE		

SAMPLING
DATE _____ S-ID#

NALYZE FOR/CODE _____

Call to Card Card Card Card Card Card Card

SAMPLING
DATE.

NALYZE FOR/CODE

SAMPLING
DATE

WALYZE FOR CODE _____

SAMPLING DATE ______

SAMPLING
S-ID#: _____ DATE _____
ANALYZE FOR/CODE: _____

S-ID# SAMPLITIG DAT ________ DAT ______

S-ID# - SAMPLING DATE

ANALYZE FOR/CODE _

GLASSWARE RELEASE CHAIN OF CUSTODY RECORD

COMPUCHEM LABORATORIES

ACCT.	NO	PROJE	CT N	IAME			ent #9		NO.			F	<u>,</u>	7	PTY 7	7	_	7	7	LED	7	O O O
AMPLE	RS: (\$1g	nature)			Rev Dat	ision/	y 30, 1989		OF CON-		5	Sep / 30 / 30 / 30 / 30 / 30 / 30 / 30 / 3			3 3	10 m			VA.			REMARKS
BOX NO.	DATE	TIME	COMP.	GRAB			ON LOCATION		TAINERS		10 1	3/	300	**/.	\$ ['] /.			400	¥0,			
					LAB	ORATO	RY GLASSWAI	RE														RELEASE ONLY
													_			<u> </u>						
	ļ	<u> </u>	$ldsymbol{f eta}$		<u> </u>																	
		<u> </u>	<u> </u>	_				~		 									ļ 		<u> </u>	
									 -				<u> </u>							 		
:		ļ	┨—				·			<u> </u>	_			_							 	
•			╂—	\vdash									-	_				-			-	
	<u> </u>	<u> </u>	╁╌		,					-	 	-	 									
	<u> </u>		╁									-	-					-	_	 	 	
		 	十一							-			 						-	 	-	
		 	1																	<u> </u>		
			1																		 	
Prepared	By: (s	ignature)			Date	Time		- <u>-</u>		Reli	inqul	shed	Ву:	(Signa	lure)		D	ale/	Time	F	lecei	ved by: (Signature)
Relinquis	shed By	y: (Signal	lure)	+	Date	Time	Received by:	(Signatur	re)	Reli	nqui	shed	By: (Signal	lure)	-	C	ate/	Time	F	lecei	ved by: (Signature)
Relinquis	hed By	/: (Signat	ure)	1	Date	'Time	Received by:	(Signatu	r +)		Dat	e/Tir	me	F	lema	rks	·	1				
•		Dis	tributi	on: Orl	ginal Acco	mpanies	Shipment; Copy to		Field Fi	<u> </u>		1_		-								
]	/3 E						ath Est					řŧ,	S alar	, ∟	ĢĒ.	<u>,</u>	ÇV.		434.2	r	* 1, . 	

Section No. 1.5 Revision No. 2 Date: May 30, 196 Page 19 of 21

DANGEROU:	S GOODS IN	EXCEPTED	QUANTITIE	S
his package contain all respects in co overnment regulati	mpliance with th	ve applicable inter	national and na	
·	Signature	of Shipper		
Title		Date		
Name and a	ddress of Ship	per		_
	ige contains sub licable box(es)	stance(s) in Class		
Class: 2	o o	់ ចំ	i i	1
and the ap	oplicable UN Nun	nbers are		Ì

:.

Attachment #11 Section No. 1.5 Revision No. 2 Date: May 30, 1989

Page 20 of 21

Shipper				Air Waybiil No. Page of Pages Shipper's Reference tumber (witerl)					
Consignee		. `	Federal Express Corporation						
Two completed and signed copies of th banded to the operator.	is Declarati	ion must b		WARNING					
TRANSPORT DETAILS				Failure to comply in all res Dangerous Goods Regula	tions may be	In breach o			
This shipment is within the . Air limitations prescribed for: (delete see-applicable)	port of De	parture		the applicable law, subject to legal penalties. This Declaration must bot, in any circumstances, be completed and/or signed by a consolidator, a					
PASSENGER CARGO AND CARGO AIRCRAFT AIRCRAFT ONLY				forwarder or an IATA care					
Airport of Destination:				Shipment type: (datab == epplicable) NON-RADIOACTIVE RADIOACTIVE					
NATURE AND QUANTITY OF DA	NGEROL	s gool	os Os						
Dangerous Goods Id	entification	n .	•						
Proper Shipping Name	Class or Divi- sion	UN or ID No.	Subsi- diary Risk	Quantity and type of packing	Packing Inst.	Authorizat			
				·					
Additional Handling Information	L !				J	 .			
•									

fled, packed, marked and labelled, and are in all respects in the proper condition for transport by air according to the applicable international Place and Date

Sinnature

Attachment #12 Section No. 1.5 Revision No. 2 Date: May 30, 1989

Page 21 of 21

	PER'S DEC	ARAT	ION	OR DANGEROUS GO	ODS	
Shipper				Air Waybill No.		
			}	Page of Pages		
				Shipper's Reference Number (aptional)		
Consignee						ı
						İ
						i I
						!
Two completed and signed to be handed to the operator	opies of this D	eclarati	on must			
TRANSPORT DETAILS				Failure to comply in all re ble Dangerous Goods		
This shipment is within the	Airport of	Departi	ıre	breach of the applicab	le law, subject	to leg
limitations prescribed for (delete non-applicable)	•		•	penalties. This Declarat cumstances, be complet	ed and/or sig	ned by
PASSENGER .:CARGO AND CARGO AIRCRAFT	7			consolidator, a forwar agent.	der or an IA	iA carg
AIRCRAFT ONLY	<u>J</u>			 		
Airport of Destination				Shipment type (delete non-ap		
NATURE AND QUANTITY O	F DANGEROUS	GOOD	S			
Dangerous Goo	ods Identification			Quantity and	Packing	1
•	Class	UN	Subsi-	tune of seeking	Inst.	Autho
Proper Shipping Name	or Divi-	or ID No.	Risk			į
		1	 			
	į	į	İ		i	į
·		į •	į		į	
	į į	i 1		i 		1
	· į	į			1	1
	į	Ì	Ì		į	İ
	İ					
				t• 		į
	1] } }		
Additional Handling Informa		<u> </u>		! !		!
Additional paneling inform	911011					
			•			

Signature

and National Government Regulations.

Section No. 1.7 Revision No. 1 Date: June 12, 1986 Page 1 of 1

Production Planning and Control SOP 1.7: Sample Custodian

The Sample Custodian has responsibility for the following tasks:

- * Raw sample storage
- * Pulling raw samples according to request list
- * Extract storage
- * Extract check-in
- * Purging extracts

* Storing extracts (final)

The Sample Custodian's first priority in performing these tasks is to ensure that sample security and chain-of-custody requirements are met. These requirements include limiting access to the walk-in and extraction refrigerator, receiving raw samples and transferring them to the extraction Lab, and recording extraction receipt and transference to the Analytical Lab. Additionally, the Sample Custodian must record any exceptions to standard handling procedures in an Exceptions Log, which documents who requested that a sample be handled differently how that sample was handled.

Section No. 1.8 Revision No. 1 Date: June 12, 1986 Page 1 of 1

Production Planning and Control SOP 1.8: Purging and Storing Extracts

SAN SAN SAN SAN SAN SAN SAN

After and EPA extract has been analyzed and the data has been reported, it is stored by case in case-specific boxes and transferred to the walk-in freezer for final storage. In the case of EPA extracts, 180 days after the report has been sent, extracts can be wither disposed of or sent back to the EPA, depending on the decision of the Deputy Project Officer.

After a commercial extract has been analyzed and the data has been reported, it is stored by CompuChem number in boxes and is transferred to the walk-in freezer for final storage.

Section No. 1.9 Revision No. 2 Date: May 1, 1989 Page 1 of 1

Production Planning and Control SOP 1.9: Handling Sample Requests

Lies that the time that the test that

With the line town their take

The Sample Request Form lists the raw samples that are to be processed by the Extraction Laboratory. The Extraction Lab Supervisor fills out and gives the Request Form to the Sample Custodian, who pulls the raw samples according to the CompuChem numbers listed on the form. The Request Form indicates the sample preparation laboratory and serves as a record of what samples were pulled for extraction on a given day. It is important from a chain-of-custody perspective that signatures and dates for the "Relinquished By" and "Received By" fields are recorded. (See example on the next page).

The Sample Custodian is responsible for maintaining a Sample Request Form Book that holds all Request Forms for future reference. Under certain rush conditions and only with the permission of the Manager of Production Planning and Control, the Sample Custodian may pull raw samples without having received a Sample Request Form. When such a request is made and approved, the Sample Custodian must record the pulling of a raw sample in the Exceptions Log, recording in the log the CompuChem Number of the sample, the date of the request, and the initiator of the request.

COMPUCHEM LABORATORIES

Internal Chain-of-Custody

多年,有明,持明,有明,是四十年四十年四十年四十年四十年

71-3

Laboratory: _			R	equested By:		
Samples For: 1 2 3 Shif (circle one)				e Requested:		
		Check Where				
EPA COMMERCIAL						
COMPUCHEM#'s	PULLED()	CONTAINERS	•	COMPUCHEM#'s	PULLED ()	CONTAINERS
1	-		11.			
			15.			
-			16.			
			15.			
-			10-			
			17.			
-			18.			
9			19.		-	
10			20.			
RELINQUISHED BY	DAT	E	RECI	EIVED BY	DATE	•
The state of the s	DAT	Έ	PECE	TVER BU		
RELINQUISHED BY	DAT	E	RECE	IVED BY	DATE	

Section No. 2.0 Revision No. 1 Date: June 12, 1986 Page 1 of 2

Production Planning and Control SOP 2.0: The Extraction Worksheet (Sample Custodian)

The Extraction Worksheet records information concerning the sample preparation processes (an example of this form and an explanation of its completion are contained in Sample Preparation SOP number 2.3.). The Sample Custodian uses this sheet to pull and check extracted samples. For the sample to leave the extraction lab, the Custodian must complete the following checks:

- * Comparison of CompuChem sample numbers appearing on worksheet to those on extract vials;
- * Match the preparation code on the worksheet to the code on the sample;
- * Check for the initiation and completion date on the worksheet;

题。(221、1861) 意义,题是一种的一种国际的一种国际的一种国际的一种国际的一种国际的

;

- * Check for the listing of a blank associated with the samples listed on the worksheet:
- * If a Quality Control Duplicate or Sample Spike is listed on the worksheet, check for the CompuChem number of the Duplicate's or Spike's original;
- * Check the Extraction Worksheet for the sample's original volume/weight and for the extract's volume/weight:
- * Check the volume of the extract vial against the volume indicated on the worksheet.

Once these checks are completed, the Sample Custodian stores samples properly and securely in the reach-in refrigerator. The Custodian then makes a copy of the worksheet, giving the copy to the Scheduling Control Clerk and the original to the Extraction Lab Supervisor.

Section No. 2.0 Revision No. 1 Date: June 12, 1986 Page 2 of 2

Initial Documentation for SOPs: Including Designated Personnel Responsibilities

This Standard Operating Procedure, Production Planning and Control, number 1.7 through 2.0, was written from an interview conducted by William J. Gargan with Bernard with Ann Marie Flaherty during the period from December 17, 1984 to January 21, 1985. The Director of Quality Assurance and the Manager, Production Planning and Control, have read and approved this procedure.

SOPs approved by: Director of Qu	Date:
SOPs approved by: Rechard Manager, Production	Date: 7/2/f6 uction Planning and Control
and Control area. If a question arise for an activity in this area, these	asks are performed in the Production Planning ses concerning the proper procedure to follow SOPs should be consulted to resolve the ques- le source of material for training purposes.
tasks has mastered these SOPs, both date this form, assuring that these s	believes the person responsible for these the manager and the employee should sign and SOPs are understood and will be followed in boratories. Please forward a copy of this urance.
Employee's name:	Date:
Employee's title:	·
Employee's name:	Date:
Employee's title:	
	Date:
Employee's title:	
Manager's name:	Date:
Manager's title:	

Long Control and Long Cons Line Cons Line Constitute Co

Section No. 1.1 Revision No. 5 Date: May 1, 1989 Page 1 of 17

1

RECTUTE TON 0 8 1989

E.

Production Planning & Control SOP 1.1: Logging In Samples

The following steps are completed for all samples as they are received by CompuChem Laboratories. (If for any reason a sample requires special handling upon receipt, the Manager of Production Planning and Control is consulted for directions as to the proper handling and documentation of the samples.)

- * Before opening and while inspecting each sample, each employee is required to wear protective clothing (lab coat and gloves). These items need to be worn at all times when in the marked areas (blue line).
- * Inspect each sample container before opening, making sure that it has not been damaged or opened during shipment. For those clients using padlocks, sealing tape, or custody seals, inspect these items to make sure that they are intact and record this observation on the chain-of-custody form (see Example 1, at the end of this SOP). If the custody seals, tapes, or padlocks are broken, contact Customer Service (for commercial samples) or the Sample Mangement Office (for EPA samples) for permission to continue processing the sample.
- * Each container is opened under the hood and checked for breakage. Check for the condition of the refrigerant (whether any ice remains or whether the cooling packs are solid) and obtain the temperature of a representative sample (liquid samples only) by immersing a clean thermometer in the sample. Record the temperature on the Sample Record (see Example 2, at the end of this SOP).
- * The temperature and pH are recorded on the log sheet. See Ex. 1 and 2 lab notices if a variance occurs.
- * Receiving personnel must sign and date all chain-of-custody documentation upon sample receipt and record any discrepancies (sample matrix, for instance) on the chain-of-custody form.
- * The Supervisor of Sample Receiving must verify that the Receiving Clerk has signed and dated the chain-of-custody form.
- * When a CompuChem SampleSaver® is received, record this receipt (on the file card) in the CLMS and insert the file card (contained in the SampleSaver®) into the file-card storage box, according to the date received.

Section No. 1.1 Revision No. 5 Date: May 1, 1989 Page 2 of 17

- * Remove samples from the shipping container and compare the sample identification information on the sample bottles to the sample information on the traffic sheets, packing lists, and chain-of-custody form included in the container (see Examples 3A and 3B, at the end of this SOP). If discrepancies exist, note the problem on the chain-of-custody form and notify Customer Service (for commercial samples) or SMO (for EPA samples).
- * Each water VOA is checked for air bubbles and headspace, and noted on the chain-of-custody form.
- * On each complete and correct <u>EPA Chain of Custody and Traffic Report</u> the statement 'Received in Good Condition' is written or stamped, initialed and dated by the receiving individual.
- * On each complete and correct <u>Commercial Chain-of-Eustody</u> the statement 'Received in Good Condition' is written or stamped, initialed and dated by the receiving individual.

'Received in Good Condition' is intended to indicate that the sample or samples were received intact with all associated sample tags (if applicable), custody seals (if applicable), pH for inorganics, and corresponding documentation in order. If there are any discrepancies in the documentation or other problems (such as breakage of the containers or chain of custody seals), the exceptions are noted on the

appropriate documents, initialed and dated.

754

- * The statement 'Received in Good Condition' does not, however, include sample temperature since EPA samples are generally received at temperatures above the recommended 4°C. The temperature is noted on the sample Log-In Sheets and the gray envelope.
- * Check incoming samples against SMO scheduled receipts (for EPA samples).
- * Log the sample in on the Accessioning Log, noting the following items:

Case number

Temperature

CompuChem sample ID

Client name or order number

Receiving date (RD)

Sampling date (SD)

Analysis codes

Matrix

Volume received

pH (Inorganics Samples Only, see PP&C SOP 3.1)

Section No. 1.1 Revision No. 5 Date: May 1, 1989 Page 3 of 17

- * For EPA samples, enter the samples' account data into the marketing section of the CLMS in order to generate the order number and associated requisition numbers. For commercial samples, contact customer service to check for the existence of the order. Then complete the order in the CLMS, and complete the EPA Scheduling Log (Example 4, at the end of this SOP).
- Enter sample into sample receipt portion of CLMS in order to generate a CompuChem number for each sample. Fill in the CompuChem number on the accessioning log sheet (this completes the log sheet).
- * A CompuChem label is generated in numerical sequence.

ř • ;

- * Label samples with the CompuChem number by wrapping each sample bottle with its computer generated CompuChem sample label. Sample labels are color coded, and are to be rotated with a different color every 2 week period by the Supervisor of Receiving or the Supervisor designee.
- * Each log sheet is reviewed by the Supervisor of Environmental Receiving to ensure information is documented. After review each log sheet is stamped as reviewed and initialed and dated.
- * Transfer the labelled samples to the secure, locked walk-in cooler facility.
- * The CompuChem number is listed on the original Chain-of-Custody next to the associated client ID when possible.
- * Access the Quiz portion of the CLMS to produce the worksheets for EPA sample analyses. For EPA samples the system will generate volatile, semi-volatile, and pesticide worksheets. For commercial and inorganic samples, pull the appropriate worksheets from the worksheet files; the analysis codes for these samples should have been included with the packing information and confirmed with customer service. Note the following destinations for the various worksheets:

Pesticide/Herbicide Worksheets: GC Lab Volatiles that do not require compositing: GC/MS Lab Inorganics: Inorganics Preparation Lab Volatiles requiring compositing, all EPA volatiles, acid/base-neutrals (commercial), semi-volatiles (EPA), and commercial TCDD's: Production Planning and Control for scheduling.

Section No. 1.1 Revision No. 5 Date: May 1, 1989 Page 4 of 17

* To produce EPA quality control worksheets for the QC samples associated with a batch of samples, access the EPA Water or EPA Solid programs of Quiz in the CLMS and enter the samples' CompuChem Numbers; copy these worksheets on green paper. To assemble commercial QC worksheets, pull the appropriate green fraction worksheets from the trays in the Shipping and Receiving area. Separate QC Sample Records are used to document the analysis of the QC samples associated with a particular batch. These are printed after the entry of sample numbers into the system and are put into green QC folders for Report Integration. Included in the commercial folder are the Sample Record (generated by the CLMS), a copy of the order form, and, if necessary a copy of the Chain-of-Custody Record.

 $I^{*}\lambda$

I that the said that the said that the

- * Assemble commercial file folders for Report Integration; include in the production sample's folder the Sample Record, Customer Sample Information Sheet and Chain-of-Custody Record; in the green quality-control folder include the QC Sample Record, which also goes to Report Integration.
- * Assemble EPA file folders for Report Integration; EPA only has the Sample Record in the file folder. A gray envelope contains all materials for the case including: yellow copy of the OTR (Organic Traffic Report), Chain-Of-Custody, original air-bill, a copy of the Log Sheet (also called Accessioning Report), a copy of the EPA scheduling Log (see Example 4A, at the end of this SOP), Custody Tags (if received) and a grey envelop contents sheet (See 4b). The white copy of the OTR is returned with a cover sheet to the EPA/SMO (Sample Management Office) (See Attachments 4c and 4d). The original EPA Scheduling Log is put in the EPA Book (kept in the Receiving area).
- * If problems arise concerning received samples, contact Customer Service (for commercial samples) or the Technical Management Staff (for EPA samples).

LABORATORY NOTICE

	- •
ì	• • • • • • • • • • • • • • • • • • • •
ب چ	LABORATORY NOTICE
-	
Š	CompuChem #
	Sample ID
	Case #
	Receipt Date
. 8	
_	The pH reading for the sample listed above was , the required pH
ż	
 ا	The Client was contacted by a member of CompuChem's Environmental Marketing Department. The Environmental Receiving Department
\	began thent was instructed to:
;	Preserve In-House by Inorganics Prep Lab
-	Analyze As Received, and Qualify with this Notice
5	Dispose - Client will resample
	T DED to Preserve
i ș	Supervisor Signature
F.	/
من _د	·· •
£.	
_	
•	. •
: 	· ·
• •	• 3 •

LABORATORY NOTICE

Sec. of The Sec.	Attachment # 2
可	•
The state of the s	LABORATORY NOTICE
6. X	
E.	CompuChem #
	Sample ID
	Case #
	Sample Type
	Receipt Date
	The required temperature for Environmental samples requiring Organic/Inorganic Analysis is 4C(+/-2C). The temperature of the sample listed above was
%	The Client was contacted by a member of CompuChem's Environmental Marketing Department. The Environmental Receiving Department was instructed to:
\$45 	Analyze As Received, and Qualify with this Notice
	Dispose - Client will resample
(2) 	
6 5	Supervisor Signature
, mete	Date
Ø	

Mi;

Phot	Phot No. POJECT NAME					O COR	· · · · · · · · · · · · · · · · · · ·	RE	العدا	io /	iour	a Tek	7//	7	الله الله الله الله الله الله الله الله			
SAMPLER	18: /3ips	-1)	,					0*	1 1					//			REMARKS	
87A, NO.	DATE	TIME	8	3		STATIO	M LOCATION	CONTAINERS	/	_	_	_	/ 	//			нешана	
				-					-		-	-	-	++				
														\Box				
														廿				
					/									士士				×
									-		_	_	-	+				Example 1
														11				
														廿				
				_					-	_	-	_	-	+				
tellingulah	ed by:	Sig-oture.		T	Doto	/Time	Received by: (Mynerous	•	Pol	nquid		N: (2)			Det	/Thme	Received by: (Myron	
winguid	ad by:	(Digradura))	+	Date	Time	Received by: (Diputure	;	Rell	nquid	had b	y: (24	 	prof	Det	/ Time	Resolved by: (Slove	
olinquid	od by:	(Signature		94: O	منابعت ومساور		Received for Laborate (Signature) Magnett; Capy to	ry by:		Des	· /Ti	me		Nomerke)			

Section No. 1.1 Revision No. 5 Date: May 1, 1989 Page 8 of 17

Example 2

Contract the long and and that have the last hand long last long long last long last

			Sample Record		
Requisition Number Case: Turnaround: Analysis Codes:		' :		CompuChem Account	Number: Number: Due:
Lab Instructions					
			Receiving Data		
Sample Identifica SS Number: Date Received: Receiving Informa Containers Receiv Matrix:	tion:	Time:	Sampling Date(s):	. SS Receiving	Code: Codes:
Deliverables Code	:		<u>Deliverables</u>		
			Lab Requirements		
	Repeat Date				
		()			
		()			
		()			
		()			·
Company Name:	_				·

Section No. 1.1 Revision No. 5 Date: May 1, 1989 Page 9 of 17

Example 2 (Lont.	le 2 (Cont.	(Co	2	е	amp	Ex
------------------	-------------	-----	---	---	-----	----

Sample Record

		•	
Requisition Number: Case:		CompuChem Number: Account Number:	
FOOTNOTES:		·	
·		•	
Applicable QA Notices:			
	•		
Company Name:			

COMPLEMENT LANGUATORIES

			CONTRACTE DISCOVER	AI UN IE	.a				
		ECEIVING Sheet for:				n. Order a.	Sheet		
	[] INCREANIC	Case ID:					[83] [83]		
	Account +:	Region e:	TAT:		Chain o	f Custody:	: [YES] [NO]		
		y:							
	COMENTS/REHARKS	SAPLE ID	CC +	120	AULYSIS	MATRIX)	VOLUME	RC	C/pH
01		<u> </u>	 	<u> </u>		<u> </u>			
02		<u> </u>	-	<u> </u>					
8		<u> </u>	<u> </u>	-			- 		
04		<u> </u>	<u> </u>	<u> </u>				_ _	
8		<u> </u>	<u> </u>	<u>i</u> _		i			<u></u>
06		<u> </u>	 	<u> </u>		1			
07		1	 	-				_	
80	 ,-	1	<u> </u>	1	1	"		- ! - !	
09			 	<u> </u>					
10	•			1	<u> </u>			1 1	! !
11	! <u> </u> 	<u> </u>		 	1			1	<u> </u>
12	! !	<u> </u>	<u> </u>	<u> </u>	<u> </u> 			1	<u></u>
12	<u> </u>		<u>i</u>	+	 				<u> </u>
13	1	<u> </u>	-	<u> </u>	<u> </u>				<u> </u>
15	1	1	 	<u> </u>	<u> </u>) 	-	<u> </u>
1 16	<u> </u>	1	1	<u> </u>			 		<u> </u>
1 17	ļ 	<u> </u>	<u> </u>	+	1				<u> </u>
18	<u> </u> -	-	<u> </u>	-	<u> </u>				<u> </u>
19	1	1	1	-		l			<u> </u>
20	!	<u> </u>	i		<u> </u>	1			!

Reviewed By:

Lugger in by:	
	(Signature)
Received By:	
	(Signature)

The state of the s

COMPLETEN LABORATORIES

Order •							Chain of Quatody (Yes) or (nc)						
	se 10:		TAT	\$50 'X: _				-	_				
	Delivered By:		Freign	: Bill •			Sec	ontract • _					
	COMENTS REMAKS	REC	SAFLE ID	α •	S	pH	Temp	Analysis Code	Matrix	Volume			
		1 1		<u> </u>		! !	! !		! !				
		1 1	· · · · · · · · · · · · · · · · · · ·	<u> </u>			!	<u> </u>	! !				
		<u> </u>	· · · · · · · · · · · · · · · · · · ·	<u> </u>		<u> </u>	<u> </u>	! }	<u> </u>				
		<u> </u>	·	<u> </u>		<u> </u>	<u> </u>	 	<u> </u>				
	! 	-		<u> </u>		<u> </u>	ļ	! 					
		<u> </u>				ļ Ļ	<u> </u>	<u> </u>					
•	<u> </u>	1 1				!		<u> </u>	1				
	<u> </u>	1 1		<u> </u>		! !	ļ	! !	<u> </u>				
<u>;</u>	<u>}</u>	1 1		ļ		<u> </u>	<u> </u>	<u> </u>		···			
)	† 	1 1		<u> </u>		<u> </u>	<u> </u>	<u> </u>					
		1 1		<u> </u>		<u> </u>	ļ Ļ	<u> </u>	1				
?	1	1 1		<u> </u>	1	! 	<u> </u>	<u> </u> 	 				
	1	1 1		<u> </u>		<u> </u>	<u> </u>	<u> </u>	1 1				
	<u> </u>			<u> </u>		<u> </u>	<u> </u>	<u> </u>	1				
5	<u> </u>	1 1		<u> </u>		<u> </u>	<u> </u>	<u> </u>	1 !				
}	! !	1 1		! !		<u> </u>	<u> </u>	<u> </u>					
7	<u> </u>	1 1		<u> </u>		1	 	<u> </u>	! ! ! !				
1	l			1 1	1		1	1	1 1				
)	1			1	1		1	1					
		1 1		1	1	1	1	1	1				

(Signature)

Section No. 1.1 Revision No. 5 Date: May 1, 1989 Page 12 of 17

Example 3A

The transfer of the transfer o

COMPUCHEM LABORATORIES

date shipped to consignee:				receiv	ved gn e e:		
number of sample: _			 				
consignee name:		_			···	·	
address: _							
- 	·						
				• 	···		
	DO	NOT	REMOVE:	FOR	COMPUCHEM	USE	ONLY

Section No. 1.1 Revision No. 5 Date: May 1, 1989 Page 13 of 17

Example 38

PLEASE NOTE THAT ANY AND ALL PRESERVATIVE(S) ARE TO BE ADDED BY THE CUSTOMER AT THE TIME OF SAMPLE COLLECTION

At the end of the sampling period, it is vital to ship the sample via express transportation. To insure proper follow-up and prompt analysis, please call 1/800-334-8525 and provide us with the following information:

- 1. Date Shipped
- 2. Time Shipped
- 3. Freight Carrier
- 4. Freight Bill of Lading Number

Section No. 1.1 Revision No. 5 Date: May 1, 1989 Page 14 of 17

Example 4A EPA SCHEDULING LOG !	FOR WEEK ENDING	SATURDAY	(DATE)		-
CASE NUMBER: NEW REGION: DELIVERABLES CODE: ACCOUNT #:	FSTIMATED SHI	PPING DATE:TURN-AROUND-	TIME: PRICE CODE:	-	
QUANTITY EXPECTED:	· ·	WATERS SEDIMENTS/SOILS			
CONCENTRATION:	: LOW	: MEDIUM	: HIGH	ł	
DATE SHIPMENT RECEIVED: QUANTITY RECEIVED:		WATERS	EMPERATURE:		
CONCENTRATION		DIOXINS			YES/NO
CONCENTRATION:	: LUW	: MEDIUM			
PROBLEMS/COMMENTS:					
SMO CONTACTED AT (TIME):		SPOKE TO			
RESOLUTION FROM SMO:			·		
					

Section No. 1.1 Revision No. 5 Date: May 1, 1989 Page 15 of 17

EXAMPLE 48

DATE:

EPA: [] ORGANIC CASE [] INORGANIC CASE [] DIOXIN CASE CASE#: NO. OF TRAFFIC REPORTS:	GRAY ENVELOPES COMMERCIAL: [] CLIENT ORDER#:	
YES NO N/A ITEM 1. [] [] CHAIN-OF-CUSTODY 2. [] [] AIRBILL 3. [] [] DAILY LOG SHEET 4. [] [] [] TRAFFIC REPORT (EPA) 5. [] [] [] SHIPMENT RECORD (EPA) 6. [] [] [] DAILY PH CHECK SHEET 8. [] [] [] SMO COVER LETTER 9. [] [] [] EPA SCHEDULING LOG 10. [] [] [] COMMERCIAL SCHED. LOG 11. [] [] [] SIS (#) COMMENTS:	COMMENTS	
SAMPLE RECEIPT/SCHEDULING:	SIGNATURES/INITALS	DATE

		DATE:	
Dear Linda,			
Reports (OTR)	the SMO and/or Regional copic for Case # ompuChem Laboratories on	•	3
If you should please do not or myself at	have any problems or question hesitate to contact Richard #219/220.	on concerning this package Bloom at extension #215	jе,
Thank you,	· —	•	
	· 		
Natalie Carte	r		
·			

1.57,

Additional Comments:

COMPUCHEM LABORATORIES SAMPLE DELIVERY GROUP (SDG) TRAFFIC REPORT (TR) COVER SEEST

Lab Name:		Contract No.: 68-01-	
Tab Code:	Case No.:	SAS No.:	
Full Sample Analysis Pri	lce in Contract: (
SDG No./First Sample in		Comple Receipt Dates	
(Lovest EPA Sample Number in first shipment of samples received under	oer .	_ Sample Receipt Date:	(MYDD/YY
Last Sample in SDG: (Highest EPA Sample Num in last shipment of samples received under	Mer	· Sample Recei <u>p</u> t Date:	(MM/DD/YY
TPA Sample Numbers in th	ne SDG (listed in	alphanumeric order):	
1			
3			
;			
*			
\$		•	
. 7			•
			
1			·
- 10		• :	•
Note: There are	a maximum of 20	field samples in an SD	
			••
	Reports to this for the order listed	orm in alphanumeric ord on this form).	er
• -			

Date

Sample Custodian

APPENDIX A.5 COMPUCHEM LABORATORIES, INC. DATA PROCESSING PROCEDURES

Section No. 8.1 Revision No. 3 Date: October 3, 1988 Page 1 of 2

8.0 Data Processing

This section summarizes the manner in which all aspects of data processing are managed and evaluated in order to maintain data integrity and characterize data quality. These processes include data collection, validation, storage, transfer, and reduction. Specific details of the procedures used by the automated data processing and computer systems operations are documented in the individual laboratory SOPs and the Report Preparation Department SOPs.

8.1 Collection

Analytical data are generated from the GC/MS computer software, GC computer, ICP computer, Atomic Absorption Spectrophotometers, Autoanalyzers, and associated laboratory instrumentation. The outputs include identifications of compounds or elements, concentrations, retention times, and comparisons to standards. Outputs are in graphic form (chromatograms), bar graph (spectra) and printed tabular form. The outputs are in standard format specified for each analysis type and are monitored for consistency. If incomplete or incorrect output is generated, corrective actions are taken according to SOPs established for each type of analysis and consistent with the manufacturer's recommendations.

All outputs of each of the instruments may be checked manually for each procedure (e.g., GC chromatographic peak area integration and calculations may be reviewed manually for baseline designation and quantitation). In the data review process (see section 8.2, <u>Validation</u>), the data produced are compared to information concerning the sample processing history, sample preparations, sample analysis, associated QC data, etc. to evaluate the validity of the results.

Section No. 8.1 Revision No. 3 Date: October 3, 1988 Page 2 of 2

Ancillary data produced for internal records and which may not be required by the customers as part of the analytical data package include the following: laboratory worksheets, laboratory notebooks, sample tracking system forms, instrument logs, calibration records, maintenance records, standard prepartion records, and associated quality control sample data. These data are available for inspection during audits to verify the validity of data and are also deliverable, depending on the client's needs.

The last the

1

A complete record of each sample's history is available for documenting its progress through the laboratory from sample receipt to reporting. Document control (see section 4.4 of the QA Plan) and chain-of-custody (see Appendix E) requirements present additional information describing these documentation and archiving processes.

Revision No. 3
Date: October 3, 1988
Page 1 of 3

8.2 Validation

THE TANK THE THE TWO THE THE

. م. ن Data validation takes on two scales. First, the Quality Assurance Department is charged with the responsibility of monitoring all laboratory QC activities, and to verify that systems are in control. These responsibilities and the manner in which they are executed are described in the QA SOPs as well as this QA Plan. The QP Department therefore plays a role in data validation in the context of the overall QA Program.

Of course, data validation also occurs on a sample-by-sample basis. This is the responsibility of the various levels of data review taking place within the laboratory. The first level of review occurs "at the bench"--this is the initial review by the instrument operator or analyst, responsible for assessing the following:

- cross-checking all sample identification numbers on worksheets, extract vials/digestate bcttles, and instrument outputs
- calculation of surrogate recoveries and internal standard responses (when applicable), and verification that QC acceptance criteria are met.
- verification that all calibration, tuning, linearity, and retention time drift checks are within QC acceptance criteria
- verification that all target analytes are within the instrument's analytical range and deciding on appropriate dilutions when necessary
- determination that peak chromatography and other instrument performance characteristics are acceptable
- verification that chain-of-custody is intact based on accompanying paperwork

The second level of review is performed by the in-lab Data Review staff. In the GC/MS Laboratory, these reviewers are all experienced Mass Spectroscopists qualified to perform mass spectral interpretation. GC Lab and Inorganics Lab Data Reviewers are degreed, senior-level chemists. The Senior Data Reviewer,

Revision No. 3
Date: October 3, 1988
Page 2 of 3

Manager of Data Review or Lab Manager also audit a percentage of these data prior to being released to the Report Preparation Department. In-Lab Data Reviewers verify all assessments previously made by the operator/analyst, and also evaluate the following:

٠.

The later than the la

2.

. . .

137

F :

- verification that all quality control blanks meet QC requirements for contamination, and that associated sample data are appropriately qualified when necessary
- calculation of matrix spike recoveries and duplicate RPDs, and verification that accuracy and precision QC criteria are met
- comparison of all injections of a sample, and comparison of matrix spikes with the original unspiked sample, for acceptable replication
- qualitative identification of all target analytes using specific SOP interpretation criteria
- verification of computer quantitation of all target analytes, including evaluation of Extracted Ion Current Profiles (EICPs) and of chromatograms for proper resolution and integration, when necessary
- verification that analytical worksheets have been completed by the operator/analyst, including date and initials
- for pesticide GC/MS or GC confirmation analyses, verification that target analyses were within retention time windows and/or evaluation of spectra for proper identification, and comparison to initial analysis
- for GC/MS analyses, evaluation of Library Search mass spectra, characterization of tentatively identified compounds, and verification of calculations for estimated concentrations of these compounds
- verification that GLP was followed relative to the correct procedure in making changes to data

The completed data package, which has been reviewed on an analytical fraction basis (i.e., volatiles, acids, base/neutrals, pesticides), is then forwarded to the Report Preparation Department. The package is then integrated with other fractions from the same sample, and with associated deliverable items as required by the client, and forwarded to the Final Technical Review staff for

Revision No. 3 Date: October 3, 1988 Page 3 of 3

the third level of review. The Final Technical Reviewer, also a senior chemist and experienced data validation specialist, assesses the complete data report (or "case", for CLP-format reports) and double-checks all items previously validated by the in-lab Data Reviewer. Additional assessments include the following:

- review of all data summary documents and verification of correct transcription from raw data
- comparative evaluation of data from individual fractions of a sample, and
 of samples from the same site, project or case, for consistency of
 analytical results and resolution of discrepancies
- checks data report or case for completeness

- for CLP-format reports, a "case narrative" must be written that authorizes release of the data, provides end-users a "history" of the sample processing, documents the quality control process used and exceptions to Statement-of-Work criteria, and summarizes any corrective actions taken.

Upon completion of all levels of review and authorization of data release by the Final Technical Reviewer, the data report (or case of reports) is sent to the Deliverables Department for mailing.

Senior members of the Quality Assurance Department are also required to audit approximately 10% of all analytical data. The QA auditor performs the same assessments as the Final Technical Reviewer. Findings from these data audits are presented in a report to management, as described in section 10.0 of the QA Plan.

Section No. 8.3 Revision No. 3

Date: October 3, 1988

Page 1 of 1

8.3 Storage

At every stage of data processing at which a permanent collection of data is stored, procedures are established to ensure data integrity and security. Specific QA Project Plans indicate how specific types of data are stored with respect to media, conditions, locations, retention time, access. The following chart indicates general guidelines as detailed in Production, Planning Control SOP 2.9:

Media	Conditions	Location	Retention Time	Access
Hardcopy	locked warehouse	off-site	client-specific	Document Custodian or other designated personnel
Magnetic Tape	locked warehouse (environment controlled)	off-site	indefinitely	Document Custodian or other designated personnel

Revision No. 3
Date: October 3, 1988
Page 1 of 1

8.4 Transfer

All data transcriptions for final reports to clients are perfomed by Report Preparation Clerks. For non-CLP reports, the reportable data is reviewed and approved by the Final Technical Reviewer, then word-processed by computer. Validation of the word-processing function is performed by a proofreader prior to release of the data. For CLP reports (whether to EPA or commercial clients), all raw data are reduced into deliverable format by Report Preparation Clerks, including the summary of data onto forms required by the Statement-of-Work. Data summaries are accomplished by utilizing a PC-based software system that extracts data directly from the laboratories' computers. The data are then sent to the Final Technical Reviewer for validation. In either case, the Final Technical Reviewer is provided with both the deliverable report and the non-deliverable back-up data, and must validate all transcription processes.

Revision No. 3 Date: October 3, 1988 Page 1 of 1

8.5 Data Reduction

原の一部で「 Mary 一層を 一層を 一層を 一層を 一般で 」ので 」の

the state of the s

Data reduction includes all processes that change either the instrument/computergenerated values, quantity of data values or numbers of data items, and
frequently includes computation of summary statistics. Documentation of the
calculation process is required. In most cases, a programmable calculator,
PC spreadsheet or computer program is used in this process. The documentation
allows the reviewer to verify the validity of the data reduction process. All
computer-generated compound lists containing the reportable results include
formulae used in performing the calculations.

CompuChem's policies regarding the use of significant figures and rounding of results are outlined in Appendix H. An extra significant figure is carried through all calculations until the final, reportable result is generated.

Analytical results are never corrected for blank (background) contamination, but are flagged and footnoted appropriately.

APPENDIX A.6

COMPUCHEM LABORATORIES, INC.

CORRECTIVE ACTION AND PERFORMANCE AUDITS PROCEDURES

Section No. 10.1 Revision No. 4 Date: October 3, 1988

Page 1 of 3

10.0 Corrective Action

10.1 Introduction

THE PART THE

1

Generally, there are two types of corrective actions that may be required when data quality falls below specified limits. The first type, and the simplest to implement and document, is corrective action required because <u>routine</u> data quality assessments are out-of-control. Surrogate and spike standard recoveries, relative percent differences between duplicates, internal standard response variations, and unacceptable blank contamination are some of these assessments in the first category. These are all performed on a sample-by-sample and/or batch basis, and corrective action is limited to evaluating the data with respect to SOP criteria, and accepting or rejecting the sample/batch. The decision that is made is clearly indicated on analytical worksheets, and unless a trend is observed during the course of data validation, additional corrective action or documentation is not necessary.

The second type of corrective action is that required when other, more global QC/QA assessments, are made. The assessments might typically indicate systematic deficiences or those affecting data useability for more than one batch (i.e., glassware contamination checks, standards preparation errors, etc.). In most cases, assessments of this nature are made by reviewing peripheral QC/QA documentation, observing procedures for comparison with SOPs or GLPs, or receiving feedback from data reviewers, management or those external to the organization (clients, auditors).

The following sections describe the QA reporting and feedback channels designed to ensure that early and effective corrective action is taken in such instances.

Section No. 10.1 Revision No. 4 Date: October 3, 1988 Page 2 of 3

In many cases, depending on the nature of the deficiency and the urgency for remedial action, a Corrective Action Report (following this section) will be completed. The report serves to document the deficiency, the required corrective action, and accountability for the action.

For observations made over longer periods of time, the QA Department issues formal summary reports to management on a monthly or quarterly basis. Following is a brief discussion of the types of reports issued to management to assess the overall effectiveness of the QA Program and to reinforce the application of Good Laboratory Practices (GLPs).

CORRECTIVE ACTION REPORT

NTE:	
OBLEM / DEFICIENCY:	
DENTIFIED DV.	
DENTIFIED BY:	
EFERRED TO:	
ORRECTIVE ACTION TO BE TAKEN:	TARGET DATE:
OLLOW-UP AUDIT FINDINGS:	•••
RESOLVED? DATE:	
SOP REQUIRED TO BE WRITTEN/MODIFIED? Y	
This form to be filed with the Quality A	

Revision No. 4
Date: October 3, 1988
Page 1 of 1

10.2 Routine QC Check Reports

| 1952 | 1952 | 1952 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953 | 1953

The following routine quality control checks (also discussed in section 9.2 of the QA Plan) are performed to verify that samples are not contaminated during transportation, preparation, analysis or storage, and that standards prepared internally are traceable to certified sources.

- -- Vendor-Supplied Glassware Checks
- -- Glassware Decontamination Checks
- -- Water Purification Systems Checks
- -- Glassware Storage Cabinet Checks
- -- Refrigerated Storage Systems Checks
- -- Reagent Purity Checks
- -- Standards Prepartion and Traceability Checks

The criteria for these QC checks and corrective action steps are detailed in the QA SOP Manual. Results are tabulated and/or plotted on control charts, and records reviewed by the QA staff. A series of quarterly reports to management summarize this information and the status of these programs.

Section No. 10.3 Revision No. 4

Date: October 3, 1988

Page 1 of 2

10.3 Monthly QA Activity Reports

The later than the later than the later than the later than the later than the later than the later than the

These reports are produced by all members of the QA staff, and summarize key QA activities during the previous month. The reports are distributed to the Director of QA, and are provided as an attachment and referenced in the Director's report to the CEO, the Executive Staff and senior laboratory management.

Included in these reports is a summary of significant quality problems observed during the period, and the corrective actions taken to remove deficiencies. The report stresses proactive measures that are being taken to improve quality or ensure compliance with QA program requirements.

Laboratory management uses the report to quantitatively measure monthly performance in terms of the number of samples processed, the frequency of repeated sample analyses due to unacceptable QC performance, and the cause of the unacceptable performance. These data are all presented in tables, Pareto control charts or attribute control charts, based on the characterization of each analysis in the Computerized Laboratory Management System (CLMS) using a system of analytical "condition codes."

The Condition Code System is used to monitor sources of data failures.

Condition code definitions are provided in an SOP to data generators and reviewers who are responsible for assigning the appropriate code to each analysis (see Appendix D). Each two-letter code is used to characterize the cause of a sample failure or the final status of the data package prior to release to the client.

Section No. 10.3 Revision No. 4 Date: October 3, 1988 Page 2 of 2

Various computer programs may be used to sort condition code data according to sample matrix and method. This system is used to pinpoint sources of error, provide feedback to management, reinforce good laboratory practices, and document laboratory performance over time. The QA staff also note in the Monthly QA Activities Report any corrective actions taken or necessary procedural changes, based on the application of condition codes.

Other items included in this report are:

- -- Summary of any changes in certification/accreditation status
- -- Involvements in resolution of quality issues with clients or agencies
- -- QA organizational changes
- -- Notice of the distribution of revised documents controlled by the QA Department (i.e., SOPs, QA Plan)
- -- Training and safety issues, if not already covered in audit reports during the period
- -- Performance of subcontractor laboratories (also communicated in separate, detailed subcontractor audit report to management)
- -- Positive feedback for acceptable performance on interlaboratory or intralaboratory tests or successful completion of audits.

Revision No. 4

Date: October 3, 1988

Page 1 of 1

10.4 <u>Laboratory Performance Reports</u>

This quarterly report presents a statistical and graphical summary of the laboratory's performance on batch-associated quality control samples analyzed over the period. Included are tables, Shewhart control charts and I-charts (for individual data points) for all surrogate and spike standard recoveries. Additionally, a monthly report to the Director of QA presents control charts and tables for all Laboratory Control Sample (Blank Spike) and Blank recoveries. The charts and tables are used primarily to document historical performance, update recovery control limits, and monitor long-range trends that might not be apparent to data reviewers evaluating data on a sample/batch basis.

Date: October 3, 1988 Page 1 of 7

10.5 Laboratory Audit Reports

Quarterly audit reports are written by a member of the QA staff and distributed to management, and summarize the results of internal laboratory Performance Audits, Systems Audits and Security/Access Audits. When external auditors are involved in Performance or System Audits, a report is written within the next week by the QA staff member coordinating the audit. The report, summarizing audit results as discussed in the debriefing as well as other observations, is distributed to the CEO and senior lab management. The report includes corrective actions required as a result of the audit, and a schedule for implementation. A follow-up audit, usually within three weeks of the distribution of this report, is conducted to verify that corrective actions have been implemented.

Performance Audits

Performance Audits are checks made by a QA staff member or other independent auditors to evaluate the quality of the data produced by the analytical system. These audits are performed independent of an in addition to routine quality control checks, and reflect as closely as possible lab performance under normal operating conditions.

These audits involve the review of approximately 10% of all analytical data reports generated by the lab for calculation and data validation procedures, and overall data quality. Errors observed during the audit are characterized as "critical" or "correctable" and tabulated. If necessary, based on audit findings, an amended data report may be sent to the customer. Following this section is a copy of the QA Audit Summary used by auditors to tabulate the data

Revision No. 4
Date: October 3, 1988
Page 2 of 7

for summary into the Quarterly Performance Audit report. A thorough discussion of these audits is included in the QA SOPs. The reports are used by laboratory managers to provide feedback to staff members and establish goals for improved performance.

!:3

A number in interlaboratory and intralaboratory tests are conducted routinely at CompuChem*, and the results are included in individual Performance Audit reports specific to each test. When new methods are available to the laboratory or new personnel are being trained, Laboratory Proficiency Tests are performed. These tests consist of quadruplicate blank spikes, containing a full complement of tests parameters to be analyzed by the method. The replicate results are analyzed by a QA staff member, who generates a summary report to the Director of QA. This report includes the standard deviation and mean recovery for each of the replicate parameters, and the data are used to statistically validate method and/or personnel proficiency. For a thorough discussion of the method validation procedures used, refer to Appendix A of the QA Plan.

On a quarterly basis, blind intralaboratory check samples are introduced into the system by the QA Department. Parameters and methods are chosen for these studies based upon independent (interlaboratory) tests from certifying agencies (including the U.S. EPA and various state agencies), Laboratory Proficiency Test results, Method Validation studies, or results from routine batch-related QC samples. The existence of these check samples in the system is known only to those personnel involved in preparing the samples and scheduling the analytical requirements into the CLMS. A thorough report, detailing the entire data generation and support functions, is completed by the QA staff and reviewed by

Section No. 10.5 Revision No. 4 Date: October 3, 1988 Page 3 of 7

the Director of QA before distribution to the CEO and senior laboratory management.

CompuChem[®] also participates in a number of external, interlaboratory performance studies. These are required as part of various agencies' certification/accreditation programs. As a member of the USEPA's Contract Laboratory Program (CLP), the laboratory is required to successfully analyze quarterly, blind proficiency samples for both organic and inorganic parameters. The CLP program also requires an annual on-site inspection by principals from the USEPA (and their contracted agents). These audits generally follow the same format described below, Systems Audits.

-

•

CompuCheme also participates in a number of state certification programs, including those for North Carolina, New Jersey, New York and Florida. All of these programs require the laboratory to submit to annual on-site inspections in order to maintain certification to perform testing on samples originating in the state. All states also require successful performance on interlaboratory check samples, submitted at least annually, though some reciprocity with the two NC programs (one for drinking water and one for wastewater certification) and USEPA-CLP is allowed under certain circumstances.

Several states utilize the laboratory's performance on the annual Water Supply (WS) and Water Pollution (WP) proficiency testing series, orginating out of the EPA Environmental Monitoring and Support Laboratory's performance on all interlaboratory and intralaboratory check samples, tabulated by parameter and method, so negative performance trends can be readily pinpointed.

Revision No. 4
Date: October 3, 1988
Page 4 of 7

System Audits

•

A System Audit is an on-site inspection and review of the QA Program for the total laboratory. While Performance Audits are a quantitative appraisal, System Audits are for the most part qualitative in nature. The System Audit may be either scheduled or unannounced before it is conducted, but occurs routinely on at least a quarterly basis. The auditor reviews the laboratories' SOPs to verify compliance with procedures and activities actually in place. Personnel and facilities are also evaluated during the System Audit. The auditor is required to investigate anything which seems in conflict with the QA Plan, the laboratory or QA SOPs, or Good Laboratory Practices.

If deficiencies are observed during a Performance Audit, and if deemed necessary, the QA Department initiates a System Audit. The audit emphasizes the actions necessary to correct deficiencies noted in the Performance Audit. A Corrective Action Report is completed, detailing all remedial actions taken, and reviewed by the Director of QA. The report must indicate the proposed implementation date and the individual(s) responsible for the action.

Many of the objectives of a routine System Audit are similar to those a client or independent auditor would hope to accomplish during an On-Site Laboratory Evaluation and Data Audit. These goals include ensuring the following:

- 1. The quality control, including necessary corrective actions , are being applied
- 2. Adequate facilities and equipment are available to perform the client's required scope-of-work
- 3. The personnel are qualified to perform the assigned tasks
- 4. Complete documentation is available, including sample chain-of-custody

Revision No. 4 Date: October 3, 1988 Page 5 of 7

5. Proper analytical methodology is being applied

the first of the f

- 6. Acceptable data handling techniques are being used
- 7. Corrective actions identified in any previous on-site visits have been implemented, and
- 8. The Laboratory Management continues to demonstrate a commitment to quality.

These objectives may be documented by completing an EPA-approved Laboratory Evaluation Checklist. In response to System Audits, any corrective actions taken are noted with reference to the auditor's deficiency report and the lab's Standard Operating Procedures.

Corriginal Status Sellar Sellar COMMENTS derestand. * 1 REJECT LOCATION/CAUSE ्र ु **ERACTION** SAMPLE NUMBER

IY A RANT JUDIT THEM

3

Missing/Incorrect:

CAM/I Calculations missing/incorrect Condition code missing/incorrect DFM/I Data footnote missing/incorrect CFM/I Correction factor missing/incorrect DWI Dry weight/percent moisture incorrect FNI Filename incorrect Form 4 missing/incorrect FFM/I LSM/I Library search missing/incorrect I/M/D QA Notice missing/incorrect RRM/I Reportable run missing/incorrect SPM/I Spectrum missing/incorrect SRM/I Sample receiving information missing/incorrect SSM/I Surrogate Summary Form missing/incorrect STM/I Standard package missing/incorrect TFM/I Tuning Form missing/incorrect

UNM/I Units missing/incorrect

WSM/I Worksheet missing/incorrect/incomplete

DAM/I OADS missing/incorrect/incomplete

Qualitative/Quantitative Errors:

HNR	Hit not reported, but should have been
HRE	Hit reported in error, should not have been reported
HAI	Hit amount reported incorrectly
CFN	Correction factor not applied to hit
SFI	Significant figures (or rounding off) incorrect
TRE	Transcription error

Miscellaneous Errors:

ISF	Internal standard area monitor indicates failure
ODI	OWA date or time incorrect
RNL	RIC not labeled
SOL	Surrogate(s) actually outside limits
WOU	Whiteout used on documents (deliverables)
CZN	Not signed off
CNI	Change not initialed

Condition Code Applications:

CS	Carryover suspected
CT	Contamination evident
RU	Repeated unnecessarily
SF	Spikes failed
UN	Unacceptable, not neede

APPENDIX A.7 COMPUCHEM LABORATORIES, INC. FACILITIES, EQUIPMENT, AND SERVICES

Section No. 6.1 Revision No. 4 Date: October 17, 1988 Page 1 of 4

6.0 Facilities, Equipment and Services

6.1 Introduction

This section describes the facilities at CompuChem®, the instrumentation and peripheral equipment, and the services provided in maintaining the facility.

CompuChemo is located in Research Triangle Park, NC, 15 miles west of Raleigh. The total facility is comprised of both the Environmental and Forensic Drug Testing Operations of CompuChem® Laboratories, Inc. The two operations have separate laboratories that function independently, including separate computer systems. Much of the office space is also separate, however, many administrative functions overlap (i.e., Accounting, Quality Assurance, Human Resources, Computer Operations) and share common office space. Facility space allocation is described in section 6.2, and includes the Environmental Operations laboratory space, Environmental office space, and administrative office space common to both operations, totaling approximately 64,000 square feet. The two operations share two adjacent buildings which are connected by a permanent, enclosed walkway. Electrical power is supplied by Duke Power Company, with a service capacity of 2000 amperes at 480 volts. The enviornmental controls for the heating, ventilating, and air conditioning systems are Honeywell Electric and provide automatic starting and stopping as well as temperature control. All critical temperature areas such as refrigerators, freezers and computer rooms are monitored 24 hours/day by an off-site monitoring firm. The temperature of the refrigerators and freezers is maintained by a standby generator in the event of a power failure. The electrical power to the computer room is regulated by a power conditioner.

Section No. 6.1 Revision No. 4 Date: October 17, 1988 Page 2 of 4

Building security is maintained at all times. The main entrance is monitored by a full-time, contracted security staff (24 hours/day, seven days/week). Visitors must sign-in at the security guard's desk and be escorted through the facility by members of the staff. The exterior doors as well as the doors of various controlled access areas within the building are equipped with electronic card readers, controlled by Rusco Electronic Card Entry Access System. A burglar alarm system has been integrated with the Rusco system to provide protection when the facility is closed. Smoke detectors, as well as associated pull stations and fire alarm horns, are provided throughout the building for fire protection. Adequate fire extinguishers and emergency equipment are also provided. The fire burglar alarms are also monitored by the off-site security firm. When an alarm sounds, the off-site personnel alert the appropriate laboratory personnel, the Sheriff's office, or the Fire Department, as necessary.

CompuChem® Laboratories contains sophisticated, state-of-the-art instrumentation and data processing equipment capable of performing most organic and inorganic analyses. Two Hewlett Packard-3000 Series 70 mainframe computers are dedicated to scheduling and tracking sample analyses through the laboratories and are directly networked to GC/MS instrumentation. An HP-3000 Series 950 mainframe provides system redundancy in the event of primary system failure, and handles additional production coordination. One of two HP-3000 Series 39 microcomputers is dedicated to systems research; the second handles all accounting functions. The Computerized Laboratory Management System (CLMS) is accessed by laboratory, marketing, systems, and accounting personnel via more than 90 CRT computer terminals.

Section No. 6.1 Revision No. 4 Date: October 17, 1988 Page 3 of 4

The Manager of Facilities and Safety, Manager of Instrumentation, and Manager of Computer Operations are primarily responsible for the evaluation, selection and maintenance of all facilities, instrumentation, and computer equipment, respectively. The Manager of Facilities and Safety is also responsible for overseeing general housekeeping services and functions as the Laboratory Safety Officer. In this capacity, the Safety Officer conducts periodic safety inspections and manages the activities of the Safety Committee.

All analytical instruments are maintained by a staff of full-time service technicians, operating during all three shifts, seven days/week (also available on-call on weekends). Instrument log books are maintained for each individual instrument in each of the laboratories (GC/MS GC, Inorganics), for recording routine maintenance performed by the operator or laboratory staff.

Additionally, service records for each instrument are kept in the Maintenance Department to record all routine and non-routine maintenance performed by service technicians.

The Pure Water Room houses a state-of-the-art water purification system. Municipal water is fed through two mixed-bed ion exchange cylinders and a high capacity activated carbon tank. The effluent is pre-polished by two mixed-bed ion exchange columns, distilled in a Corning 12-liter all-glass still, then passes through a Megapure Polishing System. This final purification process feeds water through two more mixed-bed ion exchange cartridges, and activated carbon column and a clarifying filter. Water quality is monitored daily by an in-line specific conductivity meter, and by the various method blank and instrument blank QC checks performed on the water. A similar system is used at

Section No. 6.1 Revision No. 4 Date: October 17, 1988 Page 4 of 4

an off-site warehouse facility to produce pure water used in the trip blanks that accompany SampleSavers (sample coolers) into the field during sampling operations. The Sample Preparation Laboratory and QA SOPs include additional information regarding the operation of the stills.

Two other laboratories have systems in-place to perform additional processing of the water from the Pure Water Room. Teflon transfer lines feed the water into the Inorganics Sample Preparation Laboratory and Volatile GC/MS Laboratory systems. Inorganics Lab pure water passes through an additional Millipore Pure Water System (with ion-exchange cartridges and a carbon filter), and water for the Volatile Lab is sparged with nitrogen in an all-glass reservoir for 24 hours prior to use.

The laboratory also has a full complement of support equipment and instrumentation, such as glove boxes and hoods, walk-in refrigerators, freezer units, autoanalyzers, and sonicators.

The following sections describe the laboratory area by function and equipment. The floor plan was designed to allow for the efficient and secure movement of samples and data between work areas.

Section No. 6.2 Revision No. 4 Date: October 17, 1988 Page 1 of 6

6.2 <u>Laboratory Areas</u>

Shipping and Receiving: This area is located adjacent to the laboratory section of the building. Samples arriving are identified and introduced into the scheduling and control system. The sample receiving area for environmental samples has about 1,570 square feet of floor space. The receiving area has 102 square feet of bench space for receiving and opening samples, three data entry stations, one laboratory sink and ample storage shelving.

Walk-in Refrigeration System: This area is accessed from the Sample Custodian's area outside of Sample Receiving. This 2,500 cubic feet system has two independent refrigeration units, is temperature controlled to $4^{\circ}C \pm 2^{\circ}C$ and is equipped with an activated carbon air filtering system, which maintains an environment free of organic vapors. The temperature is recorded daily. Both entrances are secured by locks and the temperature-activated alarm system is tied into a private security service. In the event of unauthorized access or temperature fluctuations, appropriate parties are notifed by the private security service. A generator maintains the temperature in the event of a power failure.

Organic Extractions and Inorganic Preparations Laboratory: This area is equipped with hoods as well as extraction equipment sufficient to process many thousands of samples per month. The environmental sample preparation laboratory has 2,008 square feet of space, four 8' fume hoods, three IEC centrifuges, two vacuum ovens, two sinks, six water baths, and 220 square feet of bench space. The air handling system for the sample preparation laboratory was custom designed for the extraction process. Conditioned 100% outdoor air is supplied

Section No. 6.2 Revision No. 4 Date: October 17, 1988

Page 2 of 6

into the room through linear diffusors and vented through exhaust ducts which extend from wall-to-wall on the north and south ends of the laboratory. This method maintains air flow at the work/stations at all times and virtually makes the room a large walk-in fume hood. A complete air exchange occurs every two minutes. Separate exhausts are provided for furnaces and hoods. Adequate cabinet space is provided. Specially-designed water baths controlled and programmable to temperature and duration are also used. The glassware preparation room has 750 square feet of floor space and is equipped with two glassware washers, 26 feet of stainless steel counters with four built-in sinks, and one 72 cubic foot annealing oven.

Solvent Storage Area: This area is accessible through a secured door adjacent to the extraction and preparation area. The room is designed with reinforced concrete walls, an automatic halon fire-extinguishing system, alarm systems and a roof that relieves pressure in the event of an accident.

GC Laboratory: The laboratory's nineteen gas chromatographs are equipped with autosamplers or purge-and-trap devices (Tekmar LSC-2) and are interfaced with a Hewlett-Packard 1000 laboratory computer for data processing (all of which are installed on a raised computer floor). A variety of detectors are attached to the GCs, including Flame Ionization (FID), Flame Photometric, Electron Capture, Thermionic Specific (also called NPD or AFID), Photoionization (PID), and Electrocoulometric (Hall) detectors.

GC/MS Laboratory: The special features included in this area are numerous. All twenty-three GC/MS systems are raised on a computer floor. This allows

Revision No. 4
Date: October 17, 1988
Page 3 of 6

gas, water, cooling and exhaust systems required to support each instrument to be introduced to the room independently, beneath the floor. There are 12 distillation units for cyanide and 8 units for phenols distillation.

Equipment is arranged in efficient work stations. In this way, specific instruments can be utilized for specific types of analyses. Several instruments are physically isolated from the rest of the GC/MS Laboratory by a glass wall (with an independent air handling system) dedicated to volatile organic analyses. These instruments are never subjected to semi-volatile work; therefore, cross-contamination of the instruments is eliminated. Furthermore, each station of instruments is staffed by experts familiar with the procedures associated with each specific method. This staffing system allows intimate daily interaction between the operator, his/her instruments and the methodologies required. All other instruments are dedicated in a similar fashion. The GC/MS Laboratory has a total of 3,380 square feet of space, and is provided with an individual power supply from a breaker panel located within the lab. The GC/MS instruments are powered by three 1-phase, 75 KVA 480/220 volt isolation transformers. Helium, the carrier gas used, is supplied from a manifold system in an adjacent room through a piping system under the raised floor. There are three of these systems, each having a catalytic scrubber to remove traces of oxygen and water, prior to entering an instrument.

The 23 GC/MS instruments are configured with both packed and capillary GC columns, and have accessories for purge and trap, direct injection, or solid probe for introduction of samples. Both electron impact and chemical ionization sources are available. Each GC/MS instrument is equipped with its own dedicated microprocessor for data processing.

~ .

Section No. 6.2 Revision No. 4 Date: October 17, 1988 Page 4 of 6

<u>Standards Laboratory</u>: This area is separated completely from all other laboratories and is equipped with its own GC instrument. Refrigeration, glove box and hood units are located in this area. The entrance to this area is locked at all times and secured by a cypher lock.

<u>Inorqanics Laboratory</u>: This area is separated completely from all other laboratories and has Inductively Coupled Plasma (ICP), Technicon autoanalyzer, Atomic Absorption Spectrophotometers (AAS) and UV/visible spectrophotometer systems. Several other analytical instruments required to perform classical analyses are also located in this laboratory. Hood systems are also an integral part of this laboratory.

Mercury is detected by flameless-cold-vapor methods established by the USEPA (Cold Vapor Technique). For maximum data management, the Inorganics Laboratory uses a mini-computer (Digital, PDP11/73) interfaced to the ICP instrument (Jarrel Ash, Model 1100).

Extract Storage: Sample extracts are stored in specially-designed refrigeration units located adjacent to the Walk-in Refrigeration System. These refrigeration units are kept locked and may be accessed only by a sample custodian. These refrigeration units are also connected to an alarm system. In the event of temperature fluctuations outside acceptable levels ($4^{\circ}C \pm 2^{\circ}C$), appropriate parties are notified by a private security service and the problem is corrected by laboratory staff.

`—

Revision No. 6.2
Revision No. 4
Date: October 17, 1988
Page 5 of 6

High Hazard Laboratory: A limited access laboratory has been designed for sample preparation aspects associated with high-hazard samples. For example, all samples requiring analysis for 2,3,7,8-TCDD are prepared in this lab. Access to the laboratory is by means of a cypher lock. The hoods are equipped with an HEPA filtration unit. Laboratory personnel use more sophisticated protective clothing than other extraction laboratory personnel (i.e. full sack suits, booties, face masks, etc).

Section 6.2 Revision No. 4 Date: October 17, 1988 Page 6 of 6

FACILITY SPACE ALLOCATION

TOTAL LABORATORY BUILDING SQUARE FEET 24,005

1.	Sample Receiving	1,570 sq. ft.
2.	Glassware Prep	750 sq. ft.
3.	Organic Extractions and Inorganics Sample Preparation	2,008 sq. ft.
4.	High Hazard Lab	450 sq. ft.
5.	GC/MS	2,840 sq. ft.
6.	Computer Room	1,450 sq. ft.
7.	Standards Laboratory	312 sq. ft.
8.	Metals (Inorganics) Instrumentation Lab	650 sq. ft.
9.	GC Lab	1,200 sq. ft.
10.	Solvent Storage	542 sq. ft.
11.	Utility	960 sq. ft.
12.	Walk-In Refrigeration System (2 units)	250 sq. ft.
13.	Miscellaneous (Canteen, Corridors, Rest Rooms, etc.)	5,000 sq. ft.
14.	Office*	6,023 sq. ft.

TOTAL PAMLICO BUILDING SQUARE FEET 55,487

1. Office* 40,142 sq. ft.

TOTAL COMPUCHEM LABORATORIES, INC. FACILITIES RESEARCH TRIANGLE PARK, NC* 79,492 sq. ft.

^{*} includes both Environmental and Forensic Drug Testing Operations.

Section No. 6.3 Revision No. 5 Date: October 17, 1988 Page 1 of 3

GAS CHROMATOGRAPH LABORATORY EQUIPMENT

Item	Model#	Serial#	CChem#	A-D#	Туре	Installed
GC GC GC	VARIAN 3700 VARIAN 3700 VARIAN 3700 VARIAN 3700	58760308-13 71280469-13 32968966-11 74550509-13	000000	2&3 7&1 23	DUAL ECD AUTOSAMPLER DUAL ECD AUTOSAMPLER FID NPD FID	1980 1980 1980 1982
GC	HP 5880	2236A04163		21	FID	1982
60000000000000000000000000000000000000	VARIAN 3400 VARIAN 3400 VARIAN 3400 VARIAN 3400 VARIAN 3400 VARIAN 3400 VARIAN 3400 VARIAN 3400 VARIAN 3400	2006 2310 2309 2312 3623 3052 2308 2307 2311	001177 001175 001178 001173 001174 001179	5 0 4 6 9 10 12 14 24	FPD ECD NPD AUTOSAMPLER ECD FID AUTOSAMPLER ECD FID AUTOSAMPLER ECD AUTOSAMPLER ECD AUTOSAMPLER ECD AUTOSAMPLER ECD AUTOSAMPLER ECD AUTOSAMPLER ECD AUTOSAMPLER ECD AUTOSAMPLER	1986 1986 1986 1986 1986 1986 1986 1986
GC	VARIAN 3400 TEKMAR LSC-2 TEKMAR ALS 0.I. 442	3053 144 1016	001357 001647	19	HALL DET PURGE AND TRAP AUTOSAMPLER	1985
GC	VARIAN 3400 0.I. 4460 HNU PI-52	3054 171-6-9B 620045	001356 001499 001362	20	PID DET PURGE AND TRAP	1985
GC	VARIAN 3400 TEKMAR LSC-2 TEKMAR ALS HNU PI-52	2306 1821 1041 620100	001176 001241 001648	18	PID PURGE AND TRAP AUTOSAMPLER	1985
GC	VARIAN 3400 TEKMAR LSC-2 TEKMAR ALS O.I. 4420	2005 1556 902 6644-5-102	000953 001316 001649	17	HALL PURGE AND TRAP AUTOSAMPLER	1985
GC	VARIAN 3400 0.I. 4460 0.I. 0.I. HNU PI-52	365-6-0020	001358 001507 001508 001509	16	PID PURGE AND TRAP LOOP SAMPLING MODULE	1985
OVEN	BLUE M SW-11TA-1	SW365	001353		OYEN	
COMP	UTER HP 1000	F1: #50.140 6V	6 3 51		ALS SYSTEM DATA PROCESSING	

CHARCOAL AIR FILTERING SYSTEM

Section No. 6.3

Revision No. 5 Date: October 17, 1988 Page 2 of 3

GC/MS LABORATORY EQUIPMENT (ENVIRONMENTAL)

OWA #	Serial#	Type Of Application	Installed
OWA - 1020	12137-0980	CAPILLARY COLUMN	9/81
OWA - 1020	12391-3-0281	CAPILLARY COLUMN	9/81
OWA - 1020	12141-0980	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12138-0980	CAPILLARY COLUMN	9/81
OWA - 1020	12140-0980	CAPILLARY COLUMN	9/81
OWA - 1020	11957-2-0180	CAPILLARY COLUMN	9/81
OWA - 1020	11957-3-0180	CAPILLARY COLUMN	9/81
OWA - 1020	11957-4-0180	CAPILLARY COLUMN	9/81
OWA - 1020		VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	11957-1279	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12391-2-0280	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12391 0281	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12139-0980	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12391-1-0380	VOA-LSC/PURGE AND TRAP	6/82
OWA - 1020	12391-4-0381	CAPILLARY COLUMN	9/81
OWA - 1020	12391-5-0381	CAPILLARY COLUMN	6/83
OWA - 1020	12645-1-1181	VOA-LSC/PURGE AND TRAP	6/83
OWA - 1020	12645-4-1181	VOA-LSC/PURGE AND TRAP	6/83
0 WA - 1020	12645-6-1281	CAPILLARY COLUMN	6/83
OWA - 1020	12645-3-1181	CAPILLARY COLUMN	6/83
OWA - 1020	12645-2-1181	CAPILLARY COLUMN	6/83
OWA - 1020	S12645-5-1281	VOA-LSC/PURGE AND TRAP	6/83
INCOS 50	13954-0387	HP-GC WITH CAPILLARY COLUM	N 1987

Section No. 6.3

Revision No. 5 Date: October 17, 1988 Page 3 of 3

INORGANIC LABORATORY EQUIPMENT

<u>Item</u>	Make	Mode 1#	Serial#	Installed
AUTO ANALYZER II	TECHNICON	TRAACS 800		1987
CIRCULATING BATH	PRECISION			1987
ANALYTICAL BALANCE	METTLER	MODEL HL 52	A76373	1980
ICP	JARRELL ASH	MODEL 1100	22483	1986
MICROPROCESSOR IONALYZER PH METER	ORION	ORION 901	93353	1979
UV VISIBLE SPECTROPHOTOMETER	VARIAN CARY	219	0438812	1981
CYANIDE/PHENOLS AUTOANALYZER	TECHNICON	AAII	GG0797940	1980
ATOMIC ABSORPTION SPECTROPHOTOMETER	INSTRUMENTATION LABORATORY	VIDEO 22(857)	2027	1987
ATOMIC ABSORPTION SPECTROPHOTOMETER	INSTRUMENTATION LABORATORY	VIDEO 22(857)	2127	1986
ATOMIC ABSORPTION SPECTROPHOTOMETER	INSTRUMENTATION LABORATORY	VIDEO 12(857)	2128	1986
VAPOR GENERATION ACCESSORY	AVA	440	1625	1986

Revision No. 4

Date: October 3, 1988 Page 1 of 7

6.4 <u>Instrument Maintenance</u>

Analytical instruments are maintained by experts employed by CompuChem® on a full-time basis. Preventative maintenance as well as major instrument repairs can be accomplished on-site. An extensive in-house stock of spare parts allows for rapid repair. CompuChem® maintains service agreements with instrument manufacturers to further assure the operational viability of all in-house equipment.

The operational condition of instruments is one of the keys to successful completion of analytical tasks. This requirement is further magnified by the necessity to complete large programmatic requirements in a limited period of time. CompuChem's commitment to instrument maintenance assures clients that equipment will be available to generate the required data.

In discussing instrument maintenance services at CompuChem®, a distinction between GC/MS instruments and other hardware is required. In the case of the GC/MS instrumentation, CompuChem® staff have full maintenance and repair responsibility. These staff have been trained by the instrument manufacturer and are fully qualified to perform the required work. For other instruments, we have service contracts for periodic maintenance visits by the vendor, although maintenance personnel do assess whether repairs can be made in-house before outside vendors are called.

All GC/MS instrument repair logs and instrument service records are maintained in individual instrument files in the instrument repair shop.

EXAMPLE 1

											CO	MPU	CHE	M S	SERV	/ICE	RE	POR	T								R-	234 A					
INSTRUMENT	NO	<u> </u>	2	DAT	E	0 7	2	3 8	6	TIME	ار	ı	5	<u> </u>		PERA	\ TO	R:		Su	son	Ba	53										
		X o	ven i	not	hoe	ting	 -	· · · · · · · · · · · · · · · · · · ·																									
START DATE	0	7	2	3		8 6	EN	D DATE		0 7	2	L	3	8	6		RE	SPO	MSI	E T	IME		1			_	5/E	Lee G	rego	ry			
START TIME	<u>L</u>		<u>_,</u>	10		11 9		D TIME				1		5	_			AL	DO	WHT	IME	L		1	2		XOB (OMPLE	TĘ?	YES	×	NO	
ASSEMBLY N	0.						t .	REV EVEL		EPA IR CODE	F/	VIL.		1		MENT NATO			P	MRT	NO	•				DE	SCR	PTION		QTY		COST	r
701	7 0	اوا	1	4 3	2			1		0 4		اه	6			F 4	•		1	Ц		Ц	1	4 4	4		use	······································		1	<u>.</u>	1	35
			1		1_						Ц					_L		Ц	L	П		Ц	_										
			1						_[_		1				1	1	Ш										1_			1
	1	1_1	_ 1_	1	1_						$\prod_{\mathbf{l}}$	_1		1	_1				1				_1_	L						1			
111	1		_1						_1			_ 1		1					1	П		Ш											1
111	1	 l l	1	1	1	1. 1			_ {					1	1	_1			l	Ш		Ш											
COMMENTS:	Ove	1 no	t he	atin	9.	24 w	elt r	olay m	ta	engeg	Ing	fo	S C	l fi	130	F4 1	blo) WIN	In	po	46	30	ppl	y.	Ro	plex	ed (use F	4,_				
	che	ked	+24	vol	ts,	che	ked	oper at	llo	n of G	x,	gev	e b	ac	k to	Оре	9 (otor	٠ د	. G.													
					_						-4																						المنسودية
OPERATOR A	CCEP	TA MC	F •								In	ATE	T	T.	,	.T •	T	T	ıΤ				Т	ŤI	MF	ī	117	101	RE VC	7290	1216)	

EXAMPLE 1 (CONTINUED)

INSTRUCTIONS

- 1. INSTRUMENT NO. LIST AS 05 FOR OWADS, 12 for OWA12, ETC. THE 4021 GC/MS/DS IS INSTRUMENT 00. ALL STANDALONE DATA SYSTEMS ARE INSTRUMENT 99.
- 2. DATE & TIME ENTER DATE AS MM/DD/YY; AUGUST 28, 1985 IS 08/28/85. ENTER TIME BY 24-HOUR CLOCK. 9:25AM IS 0925 AND 9:25PM IS 2125. THE TIME AND DATE SHOULD BE WHEN A PROBLEM IS DISCOVERED AND REPORTED VIA THIS FORM.
- 3. OPERATOR WHO YOU ARE.
- 4. PROBLEM CODE

 4 DESCRIPTION USE THE 3 DIGIT PROBLEM CODE THAT MOST APPROPRIATELY DESCRIBES YOUR PROBLEM. PLEASE DETAIL THE PROBLEM AS FULLY AS YOU CAN.
- 5. USE BLACK INK ONLY & WRITE OR PRINT LEGISLY.

PROBLEM CODES		REPAIR ACTION CODES		FAILURE ANALYSIS CODES	
P.M.	000	PIRATE PARTS	100	UNKNOWN	200
CANNOT MEET TUNE	001	ADJUSTMENT - ELEC	102	MISCELLANEOUS	202
IDOS ERRORS - List and fully	002	ADJUSTMENT - MECH.	104	OPERATOR ERROR	204
DESCRIBE WHAT THE DATA SYSTEM	1	REPLACED ASSY.	106	SOFTWARE	206
WAS DOING	1 1			HEADCRASH	208
POOR SENSITIVITY	003	RETURNED TO VENDOR REPAIR	108	MECH. DEFECT	210
DRIFTING RET. TIMES	004	RETURNED TO VENDOR WARRANTY	110	OUT OF ADJUSTMENT	29*
NO SPECTRA OR	005	REQUESTED IN-HOUSE	112	INTERMITTANT	29*
SOFTWARE	003	VENDOR SERVICE	''*	EXCESSIVE NOISE	29*
ANOMALIES	006	(NOTE P.O. #)	114	EXCESSIVE WEAR	212
GAS CHROM.	007	CLEANED SEPARATOR	116	SHORTED COMPONENT	214
DISC DRIVE	008	CLEANED MASS FILTER	118	OPEN COMPONENT	216
PRINTER	009	CLEANED SOURCE	120	FAULTY CRIMP	218
PURGE & TRAP	010	REPLACE PART	122	POOR CONTACT	220
VACUUM FAULT	011	REPAIR IN-HOUSE	124	POOR SOLDER JOINT	222
AIR LEAKS	012	UNABLE TO REPRODUCE	126	DIRTY/DUSTY	224
TERMINAL	013			LEAKING .	226
DATA SYSTEM	014			REPLACE . WITH	
CANNOT BOOT	015			1 - ELECTRICAL	
UNKNOWN	016			2 - MECHANICAL 3 - VACUUM 4 - SOFTWARE	

PREVENTIVE MAINTENANCE - 3 MONTH INTERVAL

REPAIR - PREVENTIVE MAINTENANCE CHECKS AND SERVICES GUIDE"

				SERV				
ITEMS TO BE INSPECTED	PROBABLE SYMPTOM							SERVICE INTERNAL PROCEDURE
		delly	b1-monthly	3 months	6 months		es required	NOTE: Applicable procedures are preented in the Finnign Operator Manual(s), unless otherwise specified.
Signe 3 GC								
the GC 2. Injector for packed	Inactive GC, blown fuse	 	_				×	replace fuse
columns 3. Splitless injection		 	<u> </u>				×	
for capitlary columns 4- injector septum in	obstruction, lesks		<u> </u>				×	cieen.
the GC 5. Carrier gas con-	leskage	×	L_				×	
nections/couplings							×	
6. Certier gas filter in the GC	replace when new gas cylinder is installed			•			X	
7. Filter, flow controller	dirty fliter			X				replace filter
8. Capillary column							×	Inspect or replace as
9. Packed column (glass type)	excessive usage, leaks at injection and inter-						×	needed
10. Packed column (metal type)	face port of the zone- heating block						×	† 1
11. Detector port to GC/MS Interface		 	_		-		×	†
12. GC cool down fan		<u> </u>	-	X		-	 ^	Inspect and/

These maintenance procedures meet or exceed finnigan's recommended preventive maintenance checks and services.

				SERV	ICE RVAL	•			
ITEMS TO BE INSPECTED	PROBABLE SYMPTOM		À I	E	ž	<u> -</u>	2	ERVICE INTERNAL	PROCEDURE
Hess Spectrometer	.	delly	b I-monthiy	3 months	6 months	yearly	as requi		
1. Glass jet separator	obstruction or glass	-			-	_	×	clean or replace	
2. Glass jet seperator ferrules	breskage						×	replace	
3. Mess enelyzer head essembly (in the vacuum manifold)	gross leeks, presistent pressure due to degesing of trapped gases in the						×	Inspect	
*magnet well fignge assy *CAL gas valve assy *vent valve assy	vacuum system leakage, faulty CAL gas pressure (see Pirani gauge)						XXX	Inspect Inspect Inspect	·
*water flow sensor switch 4- Quadrupole mass enalyzer	faulty switch			×			×	Inspect and/	
5. Electron multiplier	•						×	or replace	
6. Alcatel vecuum pumps (2)				×	-	_		purge weekly and replace	
7. Pfelffer turbo pump 8. Belzer turbo pump	dirty oi i			×	\vdash	-		oll	Balzer Manuel
9. Vacuum system filter/drier	excessive use, dirty filter						×	cleen & Inspect	pg23
10. Ion Source				×				replace	
*ion source filement assy *collector	isck of sensitivity, faulty peak shape,			X				clean, inspect or replace as	
"lens "eperture "lon volume	no autotune	w11	h ev	T Y	file	enti ment emen	•	required	
GC/MS Interface Oven									
1. Capillary interface tubing	p lugged						×	clean, Inspect	
2. Separator divert fitting 3. Yacum divert valve				×		-	×	place	
							×		
Power Module									
1. MS power supply 2. Turbo power supply				×			×	measure & verify PCB	
Card Cage Module				×			×		
1. Air filter at bottom of cage				×				clean and/or	
2. Fan	of air flow burned out fan			×			×	replace	
3. Signal cable on Digital 1/0 PCB					-		×	Inspect for secure fit	

REPAIR - PREVENTIVE MAINTENANCE CHECKS AND SERVICES GUIDE (Cont.)

	•			ERV	ICE RVAL	•			
ITEMS TO BE INSPECTED	PROBABLE SYMPTOM		th by	£	ş		required	SERVICE INTERNAL	PROCEDURE
		delly	b !-month! y	3 months	6 months	yealy	85 TO	•	
Nova Computer					-	-		Inspect end/ar	
1. Fan	faulty fen rotation			×	-	-		reprece	
Perkin-Einer Disk Drive				Ĥ		\vdash			·
1. Output signal		 	-	×				check and varify	ł
2. Adjustable DC voltages (+5V, +15V, -13V)				×				·	
3. Brushes								cleen end7 or replace	
4. Positioner cerriege guide	Ì				×			clean and Inspect	ļ
Talls 5. Spindle chuck end cone					×			cleen end Inspect	P/ECEM Manual
6. Read-urite heads				x				Inspect and repair	P/ECEN Menuel
7. Fixed disk				x]
8. Air ffit u						X			P/ECEM Menuel
*profilter *main filter				X				replace replace	Ĭ
9. Slower ground brush						x		replace	
10. Spindle ground brush	1					×		replace	
11. Blower drive belt								replace	P/ECEM Manual

COMPUCHEM LABORATORIES

PROJECTED PREVENTIVE MAINTENANCE SCHE:

FINNIGAN GAS CHROMATOGRAPH / MASS SPECTROMETER

		JANUARY	1990	TO JU	NE 1990			
UNIT #	NORMAL ROUTINE	DUE FROM PREVIOUS	JAN	FEB	MAR	APR	MAY	Jun
01	04-08-12					DUE		
02	03-07-11				DUE			
03	01-05-09		DUE				DUE	
04	03-07-11				DUE			
05	03-07-11				DUE			
06	01-05-09		DUE				DUE	
07	02-06-10			DUE				DUE
08	02-06-10			DUE				DUE
09	04-08-12					DUE		
10	04-08-12					DUE		
11	02-06-10			DUE				DUE
12	02-06-10			DUE				DUE
13	01-05-09		DUE				DUE	
14	01-05-09		DUE		•		DUE	
15	04-08-12					DUE		
16	01-05-09		DUE				DUE	
17								
18	03-07-11				DUE			
19	03-07-11				DUE			
20	03-07-11				DUE			
21	02-06-10			DUE				DUE
2 2	04-08-12					DUE		
23	02-05-10	•		DUE			13	DUE

Revision No. 0
Date: October 17, 1988
Page 1 of 3

6.5 Material Procurement and Control

CompuChem's Purchasing Department has two prime objectives: (1) maintain sufficient supplies of all required items as needed, and (2) encourage all forms of competition in order to aggressively seek the best total value in a combination of supply, price, required quality, and service.

Procedures for Purchasing Materials: Department and laboratory managers have primary responsibility for maintaining adequate inventory of supplies and ensuring that all supplies/equipment meet or exceed quality requirements.

Managers work through the Purchasing Department to accomplish these objectives.

CompuChem® uses competitive inquiries or requests for bids, along with appropriate negotiation, to provide equal opportunities for potential and current suppliers to earn CompuChem's business and to allow the laboratory to seek the best total value. Long-term considerations include reliability, price, required quality and service. Vendors are encouraged to bring to CompuChem's attention new or improved materials, equipment and services. Suppliers must maintain the confidentiality of competitively sensitive information which is obtained from the Purchasing Department or other CompuChem® personnel. Prices and related information, whether accepted or not, will not be disclosed.

Each year, various vendors will supply the laboratory with solvent/chemical samples during the bidding process. The laboratory evaluates each vendor's sample, as described in the next section, before the bid is considered by the Purchasing Department. If solvent/chemical quality is equivalent, then price and service are considered. Prices are kept low because of the highly competitive market and the high volume used by the laboratory.

Revision No. 0.5

Date: October 17, 1988

Page 2 of 3

Material Quality Inspection: Managers interact with the Quality Assurance Department when purchasing supplies/equipment that could potentially affect data quality, and therefore testing prior to use in the laboratory. The Director of QA (or designated QA staff member) determines the appropriate test procedures and evaluates the resulting test data. A similar validation process is used in testing new instrumentation, as described in Appendix A. All new lots of standards, reagents, and glassware are tested as described in section 9.2 of the QA Plan and in the QA SOP Manual. The quality testing of solvents and other high-volume chemicals is described further in the following section.

Additionally, the laboratory continually evaluates the integrity of these materials by performing the routine QC method blanks with every sample batch as described in sections 9.2 and 9.3 of the QA Plan.

When variability is exhibited in the quality of vendor-supplied materials or services, the laboratory/department manager is responsible for working with the Purchasing Department to find a suitable alternative.

Chemical and Standard Inventory Procedures: All chemicals other than organic standards are inventoried by appropriate laboratory manager and re-ordered as needed, with adequate time allowed for order processing, shipment and quality testing. The vendor supplying extraction solvents first provides a test sample from a particular solvent lot. After testing by the laboratory, if the lot is approved, several cases of the lot are purchased, with the remaining cases of the same lot stored by the vendor. The vendor is responsible for keeping inventory of the solvent lot, and when only a few cases remain, provides the laboratory with a test sample from a new lot. The process is repeated so that

Date: October 17, 1988
Page 3 of 3

a second approval lot is immediately available once the first is consumed. The lab manager maintains files of all test data to verify solvent purity. All extractions are traceable to the approved solvent lot used in sample preparation.

Organic standards are prepared internally in the Standards Laboratory, and the Standards Laboratory Chemist is responsible for maintaining adequate inventories and initiating standard purity testing and tests of each standard preparation lot, as described in section 9.2 of the QA Plan and in the QA SOPs.

Solvent Storage and Waste Disposal: All solvents in use in the laboratory are kept in solvent storage cabinets, which are vented and specifically designed for this use. Acids are kept in a separate, specially designed storage cabinet. Solvents and other chemicals not in use are stored in the Solvent Storage Room, described in section 6.2. Periodically, a waste disposal firm removes the laboratory's waste, stored in 55-gallon drums, to a licensed hazardous waste landfill.

Section No. 6.6 Revision No. 0 Date: June 6, 1989 Page 1 of 2

6.6 Glassware Preparation/Decontamination

Laboratory Glassware: Glassware used in the Organic Sample Preparation

Laboratory is washed initially by hand in a hot, nonphosphate, laboratory-grade detergent solution, then loaded onto racks and into a stainless steel industrial dishwasher. When the normal wash cycle is completed, the rinse cycle is started and the glassware is rinsed continuously (approximately 5 minutes) with ordinary tap water. This is followed by a second rinse cycle (approximately 5 minutes) using laboratory-pure (distilled, deionized) water. The rack is then removed from the dishwasher, loaded onto aluminum trays and annealed at 500°C for six hours. Glassware used for Inorganic Sample Preparation and volumetric glassware, such as pipets and flasks, are not annealed but instead air-dried while inverted. After annealing or air-drying, the decontaminated glassware is loaded onto large carts which are labeled and segregated from unprepared glassware. Inorganics glassware is loaded onto a clean Inorganics Station (cart) and distributed directly to the lab.

SampleSaver® Glassware: SampleSaver® glassware is prepared separate from all other laboratory glassware, and the procedures differ slightly, depending on the type of container. One-liter glass bottles, depending on their condition, may be recycled. In such cases, they are washed, rinsed and dried as described above, but separate from all other glassware. After this step, they may be mixed with unused glassware and the procedure repeated as described above for laboratory glassware. The glassware is baked at 230-260°C for one hour, then loaded onto clean carts for cooling. Once cool, they are capped, loaded into boxes (those used for shipping the unused containers from the vendor to the lab) and stored for later use.

Section No. 6.6 Revision No. 0 Date: June 6, 1989 Page 2 of 2

For 500 ml plastic containers used for metals and Mercury sample collection, the containers are soaked for one hour in a 50% nitric acid solution following the soap solution wash. After draining, the containers are rinsed three times with tap water, then three times with laboratory-pure water. The bottles are inverted and air-dried, capped, then stored in the Inorganics SampleSaver® cabinet in the Glassware Preparation area.

Glass bottles used for volatiles, Cyanides and Phenols sample collection (40 ml) are prepared in the same manner as the liter containers described above, but are baked at 260°C for one hour. The bottles are removed from the oven as soon as they are cool enough to touch, then capped immediately with septum-sealed, Teflon-lined caps and stored in a contaminant-free cabinet.

Plastic caps, Teflon discs (cap liners) and Teflon-lined septa are washed and rinsed in the same manner as the glassware above, but are baked for one hour at 105°C. Following a brief cooling period (cool enough to touch), the discs and septa are placed into their caps and used to seal the cleaned liter and 40 ml bottles.

APPENDIX A.8

COMPUCHEM LABORATORIES, INC.

SOP MODIFICATIONS AND SPECIAL CONSIDERATIONS



SOP MODIFICATIONS AND SPECIAL CONSIDERATIONS ERM/ECC PROJECT

CompuChem Laboratories, Inc.
March 18, 1992

Modifications to OLM01.0 SOW (3/90 Organic CLP, with Revisions):

Based upon recent laboratory MDL studies, the cleanup objectives for bis(2-ethylhexyl)phthalate can be met by additional concentration of the SV extract to a final volume of 0.5 ml. To maintain the SOWspecified on-column surrogate standard concentration, one-half the normal surrogate volume will be added to the sample immediately prior to extraction. The associated method blank will be extracted in the same manner to monitor background contamination (which is expected to be elevated due to the additional concentration factor). are known to be common field and laboratory contaminants, and represent potential method interferences of concern with this approach. The SOW stipulates that the maximum allowable concentration of bis(2-ethylhexyl)phthalate in a method blank (with a final concentration of 1.0 ml) is 5 times the CRQL, or 25 ug/L. Because of the special interest in the phthalate, and based upon historical and anticipated background levels, these criteria will be lowered to 5 ug/L. If these criteria are not met, however, associated samples will only be reextracted and reanalyzed if this can be accomplished within the regulatory holding time requirement.

The cleanup objective for isophorone can be met without modification to the 3/90 SOW analytical methods. Since this compound is analyzed in the same fraction with the phthalate, however, the MDL will also be lower by a factor of two. The Form I's for both isophorone and bis(2-ethylhexyl)phthalate, when not detected, will be modified such that 1/2 the MDL is reported (with the standard "U" flag) in place of the CRQLs. The "J" flag will not be applied to these two compounds since the MDL is being referenced rather than a contract-mandated quantitation limit. By definition, results below the MDL (or in this case, 1/2 the MDL) are indistinguishable from background noise.

The detection limit to be reported (without correction for dilutions, etc.) for bis(2-ethylhexyl)phthalate is 1.3 ug/L (the MDL is 2.52 ug/L). The detection limit for isophorone will be reported at 1.3 ug/L (the MDL is 2.54 ug/L). All other compounds will be reported with 1/2 the normal SOW-specified CRQLs.

This procedure will be documented by reference, and as an attachment, to the case narrative.



Modifications to ILM01.0 SOW (3/90 Inorganic CLP, with Revisions):
Based upon recent laboratory MDL studies, the cleanup objective for cyanide can be met without modification to the SOW analytical methods. The Form I, when cyanide is not detected above the MDL, shall be modified so that the MDL of 0.8 ug/L is reported with the "U" flag (there is no IDL for cyanide). The CRDL is not considered when reporting or qualifying these data. Forms III, V, VI and X will also be changed to reflect the cyanide MDL.

Due to limitations in the software (which has been "hardwired" to prevent entry errors and contract non-compliances), the modification to the detection limit and corresponding data flags may require hand-corrections, which will be dated and initialed. If this is the case, electronic data (diskettes) will not contain the manual entries which are modifications to the SOW diskette format requirements. If programs can be conveniently altered and validated, the hardcopy data may not require hand-correction and the diskette data will reflect the correct MDL for cyanide.

This procedure will be documented by reference, and as an attachment, to the case narrative.

Modifications to OLC01.0 SOW (6/91 Low Concentration Organic CLP, with Revisions):

Aqueous volatile samples are to be analyzed following the 6/91 SOW for Low Concentration Organics. The SOP is amended for this project with the following modifications:

A second aliquot of the sample is to be analyzed (from a previously un-opened vial) by Method 8010 in order to achieve the cleanup objective of 0.38 ug/L for 1,1-dichloroethane. The 6/91 reporting forms are to be used, and the 8010 MDL for 1,1-DCA (0.35 ug/L) is to be reported with the "U" flag for non-detects. The 8010 result for 1,1-DCA is to be manually reported on the 6/91 Form I if the concentration is between the GC MDL of 0.35 ug/L and the 6/91 CRQL of 1.0 ug/L. A concentration above 0.35 ug/L will be reported without the "J" flag even if this value is still below 1.0 ug/L. Results for 1,1-DCA greater than or equal to 1.0 ug/L will be reported from the GC/MS 6/91 analysis. In such cases, mass spectral data will be provided to confirm identification of this compound

Both GC/MS and GC 8010 results will be reported in all cases.

Note that due to lack of homogeneity in some sample matrices, poor method precision near the detection limit, and variations in sample collection, GC and GC/MS results may vary. The laboratory will not repeat sample analyses solely due to minor variations between the GC and GC/MS results.



The cleanup objective can be met for 1,1,2-trichloroethane and tetrachloroethene without modification to the 6/91 analytical methods. The reporting requirements will be modified such that the MDLs will be reported for these two compounds rather than the CRQLs. The MDL for 1,1,2-trichloroethane is 0.4 ug/L, and the MDL for tetrachloroethane is 0.6 ug/L.

Since results will not be reported below the MDL for any of these three compounds, the "J" flag will not be applied. The "J" flag will be applied to all other 6/91 compounds if the concentration is between one-half the CRQL and the CRQL, so long as mass spectra meet qualitative identification criteria.

A MS/MSD will be performed for the 6/91 aqueous volatile samples in addition to the SOW-specified LCS. The MS/MSD will be spiked with the 3/90-specified target compounds (toluene, chlorobenzene, benzene, 1,1-dichloroethene, and trichloroethene) at a concentration of 10 ug/L on column (10 ul of a 50 ug/L standard). The 3/90 advisory spike recovery and RPD criteria will apply, but will not be used as the sole basis to determine whether or not reanalysis is required (as indicated in the SOW). The MS/MSD will not be repeated if the LCS meets all 6/91-specified QC acceptance criteria (unless the MS/MSD has been prepared or analyzed incorrectly). The LCS is used as evidence of sample matrix effects in the MS/MSD and associated samples. The MS/MSD will be manually reported on a 3/90 Form III; only non-spiked compounds will be reported on the associated Form I's for the MS/MSD (consistent with the SOW).

The method-specified MS/MSD will also be analyzed and reported for each 8010 batch, along with all other required deliverables. No SOP modifications are required for the 8010 analysis.

This procedure will be documented by reference, and as an attachment, to the case narrative.

Minimization of Dilutions:

The laboratory Standard Operating Procedures and guidance established by the SOWs are to be followed, without exception, with regard to sample screening and dilution:

If the sample is screened and it is determined that concentrations are such that instrument damage or detector saturation may result, the sample is diluted. In some cases, due to sample viscosity or matrix, it will not be possible to extract, purge and/or inject the sample without dilution. If this is not a problem, and the screen indicates that a low level dilution or neat (undiluted analysis) is in order, then the sample is to be analyzed accordingly.



If after this analysis, there are high concentration analytes outside the instrument's analytical range (highest multipoint standard concentration), the sample is to be further diluted until the high level analytes are in the upper half of the analytical range. If the lower level analytes present in the initial run are not diluted out, then only this final dilution is to be reported. If, however, some TCL analytes are lost in this final dilution, then the low level dilution/neat analysis is also to be reported and is billable.

Again, if the sample cannot be analyzed without dilution due to sample matrix/composition considerations, then a low level dilution/neat analysis will not be available. The laboratory shall make every attempt to analyze the project samples, following our usual SOP, with minimal dilutions.

APPENDIX A.9

COMPUCHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURE FOR VOLATILE ORGANICS IN SOIL ANALYSIS

INTRODUCTION TO THE STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF VOLATILE ORGANICS IN SOIL

CompuChem Laboratories' sample preparation and instrument procedure SOPs for the analysis of volatile organics in soil are included in this Appendix. The following is some additional project-specific information regarding these SOPs.

- 1. Instrument procedure number 282 will be used for this project.
- 2. The list of deliverables is provided on the last page in this Appendix, at the end of the instrument procedure SOP.
- 3. The QA/QC requirements for this analysis are presented in Table 5-2 and its attachments, included at the end of the instrument procedure SOP.

Section No. 3.1.1.6 Revision No. 0 Date: 6-1-87 Page 1 of 2

SAMPLE PREP PROCEDURE 3.1.1.6: -177 3rd Edition Solid Low Level Volatiles in Soils. Sediments, and Sludges

SUMMARY OF METHOD

A five (5) gram portion of a soil, sediment, or sludge sample is weighed into a graduated impinger. The impinger is sealed with a glass stopper and placed in the GC/MS refrigerator (4°C) for analysis.

PROCEDURE

- 1. Glassware must be scrupulously clean. The glassware components used in this procedure are: graduated midget impinger bottles and 24/40 stoppers. The impingers and stoppers are washed with hot soapy water, rinsed with hot water and finally rinsed with laboratory pure water. The impingers and stoppers are dried at 110°C for one (1) hour or overnight in the Grunberg oven. If the impingers and stoppers have been stored in a laboratory environment, place in oven for one (1) hour (110°C) and allow to cool in a contaminant-free environment before using. Then rinse the glassware thoroughly with laboratory pure water before use.
- 2. When the samples are ready to be prepared, assemble the following items in the designated hood: a clean spatula and a top-loading Ainsworth balance.
- 3. For each sample; label (with permanent ink on tape) an impinger vessel with the CompuChem® number of the sample, the date and the procedure number.
- 4. Remove the ground glass stopper from the impinger and place on a Kimwipe.
- 5. Place the impinger on the Ainsworth balance and tare.
- 6. Remove the top from the bottle containing the soil/sediment/sludge sample. Mix the sample thoroughly.
- 7. Transfer 5 grams \pm 0.05 gram of the sample to the impinger using a spatula. Record the sample weight on the appropriate worksheet to one significant figure to the right of the decimal, i.e. 5.0 grams. Do not transfer twigs, stones, etc. when weighing the sample.
- 8. After the sample has been weighed, replace the ground glass stopper. An adequate seal is made by slightly twisting the stopper into the impinger. CAUTION: Care should be taken to exclude any soil/sediment/sludge particles from the ground glass portion of the impinger since particles freeze the stopper when twisted onto the impinger.

Section No. 3.1.1.6 Revision No. 0 Date: 6-1-87 Page 2 of 2

- 9. Store the prepared sample in a wire rack until the complete set is weighed.
- 10. A clean spatula should be used for each weighing.
- 11. Repeat steps #3-10 until the required number of samples are prepared, completing each worksheet as the preparation is accomplished. When the set is complete, store the samples in the GC/MS laboratory refrigerator.

Standard Operating Procedure (SOP) Documentation Form

Standard Operating Procedures (SOPs) describe in detail how tasks are performed in specific areas.

Because they are used for training as well as for legal documentation, it is important that SOPs reflect the most current practices of the laboratory or department. In turn, we must keep careful records of who wrote or revised SOPs, when they became effective, and when it is time for SOPs to be reviewed. This form must accompany all SOPs to help us record that information.

Please be sure that the shaded area of this form is completed before you give the new or revised SOP to Quality Assurance for approval.

Procedure prepared by:	
Proceeding approved by a least to announce of the process of the p	era Lesas
	·.
Procedure approved by Quality Assurance Representative:	Dete: 3/21/92
7	Dates
Procedure received by Technical Communications: Swame John	3/20/92

3/19/93

Annual Review Date:

Revision No. 4.1.23
Revision No. 3
Date: March 17, 1992
Page 1 of 23

<u>Instrument Procedure SOP 4.1.23</u>: Method 8240 3rd Edition Solid and All Subsets

1.0 Scope and Application

This Standard Operating Procedure (SOP) is used for each of the 8240 EPA 3rd Edition Solid instrument procedures and all of their subsets. The following list relates instrument procedure codes to analysis type:

Instrument	Procedure	Analysis		
282		8240 3rd Edition Solid and Library Search		
413		EPA CLP-Low Level Solid		
288		3rd Edition Full List Low Level Solid		
265		Comm. HSL VOA; Low Level Solid Case ID'd		
291		CLP + 1,2,3 Trichloropropane Low Level Solid		
285		CLP + CL3F-Methane Low Level Solid		

2.0 Summary

This method is for the analysis of 8240 3rd Edition solid samples, with and without library search, COMM HSL VOA; Case ID'd; CLP + 1,2,3 Trichloropropane, CLP + CL3F-Methane Low Level Solid and EPA CLP Low Level Solid samples. The working linear range of measurement is up to 200 ppb except for acrolein, crotonaldehyde, and acrylonitrile (2000 ppb). All standards, blanks, samples, and other required runs must be injected within twelve hours following the injection time of BFB. All injections must be recorded on the instrument log along with date, time (use a twenty-four hour timetable), volume injected, operator ID, and any comments relevant to the injection. Upon completion of a tune, unused run times must be crossed out by the chemist and the log name subsequently signed by the chemist. The blue copy remains with the tune package while the yellow (bottom) copy is filed in the log book for that instrument.

3.0 Apparatus and Materials

3.1 Purge-and-trap device--The purge-and-trap device consists of three separate pieces of equipment: the sample purger, the trap, and the desorber. Several complete devices are commercially available.

Section No. 4.1.23 Revision No. 3 Date: March 17, 1992 Page 2 of 23

- The recommended impinging chamber is designed so that 5 ml of lab pure water can be added to solid samples. The gaseous headspace between the water column and the trap must have a total volume of less than 15 ml. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The needle sparger is used because it provides equivalent performance to the purge device described in SOP 4.1.22 for the 8240 3rd Ed. liquids.
- 3.1.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. Starting from the inlet, the trap must contain the following amounts of adsorbents:

1/3 of 2,6-diphenylene oxide polymer,

1/3 of silica gel,

1/3 of coconut charcoal.

It is recommended that 1.0 cm of methyl siliconecoated packing be inserted at the inlet to extend the life of the trap. If it is not necessary to analyze for dichlorodifluoromethane or other fluorocarbons of similar volatility, the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap.

If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap. Before initial use, the trap should be conditioned overnight at 180°C by backflushing with an inert gas flow of at least 20 ml/min. Vent the trap effluent to the room, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 minutes at 180°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

Traps normally last 2-3 months when used daily. Some signs of a deteriorating trap are uncharacteristic recoveries of surrogates, especially toluene-dg; a loss of the response of

Revision No. 3. Date: March 17, 1992
Page 3 of 23

the internal standard during a 12-hour shift; and/or a rise in the baseline in the early portion of the scan.

- 3.1.3 The desorber should be capable of rapidly heating the trap to 180°C for desorption. The trap bake-out temperature should not exceed 220°C.
- 3.1.4 Trap Packing Materials
 - 3.1.4.1 2,6-diphenylene oxide polymer--60/80 mesh, chromatographic grade (Tenax GC or equivalent).
 - 3.1.4.2 Methyl silicone packing--OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.
 - 3.1.4.3 Silica gel--35/60 mesh, Davison, grade 15 or equivalent.
 - 3.1.4.4 Coconut charcoal--Prepare from Barnebey Cheney, CA-580-26 lot #M-2649 by crushing through a 26 mesh screen (or equivalent).
- 3.2 Heat block--Should be capable of maintaining the purging chamber to within 1°C over the temperature range of ambient to 100°C.
- 3.3 Gas Chromatography/Mass Spectrometer/Data System
 - 3.3.1 Gas chromatograph—An analytical system complete with a temperature—programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation. For some column configuration, the column oven must be cooled to < 30°C; therefore, a subambient oven controller may be required. The capillary column should be directly coupled to the source.
 - 3.3.2 Gas chromatographic column--75 m \times 0.53 mm ID capillary column coated with DB-624 (J&W Scientific), 3- μ m film thickness, or equivalent.

Section No. 4.1.23 Revision No. 3 Date: March 17, 1992 Page 4 of 23

- 3.3.3 Mass spectrometer—Capable of scanning from 35 to 300 amu every 2 seconds or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for Bromofluorobenzene (BFB) that meets all of the criteria of BFB when 50 ng of the GC/MS tuning standard (BFB) is injected through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.
- 3.3.4 GC/MS interface--The GC is interfaced to the MS with an all-glass enrichment device and an all-glass transfer line.
- 3.3.5 Data system -- A computer system that allows the continuous acquisition and storage on machinereadable media of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the mass spectrometer. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. most recent version of the EPA/NIST Mass Spectral Library should also be available.
- 3.4 Microsyringes--10-, 25-, 100-, 500-, and 1,000- μ L.
- 3.5 Syringe valve--Two-way, with Luer ends (three each), if applicable to the purging device.
- 3.6 Syringes--4-, 10-, or 25-ml, gastight with shutoff valve.
- 3.7 Balance--Analytical, 0.0001-g, and top-loading, 0.1-g.
- 3.8 Glass scintillation vials--20-ml, with Teflon lined screw-caps or glass culture tubes with Teflon lined screw-caps.
- 3.9 Vials--2-ml, for GC autosampler.
- 3.10 Disposable pipets--Pasteur.

Revision No. 3
Date: March 17, 1992
Page 5 of 23

- 3.11 Volumetric flasks, Class A--10-ml and 100-ml, with ground-glass stoppers.
- 3.12 Spatula -- Stainless steel.

4.0 <u>Interferences</u>

- Volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap are major contaminant sources. Avoid using nonpolytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components because such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted. If reporting values not corrected for blanks results in what the laboratory feels is a false positive for a sample, this should be fully explained in text accompanying the uncorrected data.
- Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. To prevent this you should rinse the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After analysis of a sample containing high concentrations of volatile organic compounds, one or more calibration blanks should be analyzed to check for cross contamination. samples containing large amounts of water soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the whole purge and trap device may require dismantling and cleaning. Screening samples prior to purge and trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique or by Method 3820 (Hexadecane Extraction and Screening of Purgeable Organics).

Section No. 4.1.23
Revision No. 3
Date: March 17, 1992
Page 6 of 23

4.3 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing.

Laboratory clothing worn by the analyst should be clean because clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

4.4 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

5.0 Safety

- 5.1 The toxicity on carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Preparation of calibration standards and samples are perfomed in a fume hood to minimize any risk.
- 5.2 The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,2-dichlorethane, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethane, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood.

6.0 Order of Analysis

6.1 BFB

All criteria must be met according to requirements established by the EPA. GC/MS tuning and Mass Calibration forms must be printed out and attached to the instrument runlog. Relative abundances are calculated to two decimal places.

Section No. 4.1.23
Revision No. 3
Date: March 17, 1992
Page 7 of 23

	BFB KEY IONS AND ABUNDANCE CRITERIA		
Mass	Ion Abundance Criteria		
50	15.0 - 40.0 percent of the base peak		
75	30.0 - 60.0 percent of the base peak		
95	base peak, 100 percent relative abundance		
96	5.0 - 9.0 percent of the base peak		
173	less than 2.0 percent of mass 174		
174	greater than 50.0 percent of the base peak		
175	5.0 - 9.0 percent of mass 174		
176	greater than 95.0 percent but less than 101.0 percent of mass 174		
177	5.0 - 9.0 percent of mass 176		

6.2 Initial Calibration

An instrument without a valid initial calibration for this analysis or valid calibration check standard not meeting all required criteria requires an initial calibration. The initial calibration must meet all criteria as established by the EPA. After obtaining a valid initial calibration, a valid calibration check standard must be obtained before starting any sample analysis.

6.3 Calibration Check Standard

If the instrument has a valid initial calibration and the calibration check standard meets all requirements (SPCC and CCC compounds as established by the EPA), then it may be used for sample analysis. It is performed within each BFB 12-hour tune period and analyzed immediately after the BFB.

6.4 Instrument Blank

A valid instrument blank must be obtained before analysis of any sample. For a definition of a valid instrument blank see Section 8.0 on instrument blanks.

A valid blank must be obtained before sample analysis can take place.

Section No. 4.1.23
Revision No. 3
Date: March 17, 1992
Page 8 of 23

6.5. Samples

All samples must be injected within 12 hours of BFBs' injection time. Samples should be analyzed according to batch and due date. Other required injections such as quarterly proficiency tests, sample spikes, blank spikes, etc. must also be analyzed during this time.

6.6 Initial Calibration

6.6.1 Frequency

An initial calibration must be performed if the instrument does not have a valid initial calibration for this method or if the calibration check standard fails to meet all require criteria (established by EPA).

6.6.2 Nominal Concentration Values

The nominal concentration values and standard IDs for the initial calibration are as follows:

Standard ID	Concentration (µg/1)	
1910	200	
1909	150	
1908	100	
1907	50	
1906	20	

6.7 Standards Preparation

Standards are prepared for any given level by using the volumes listed below. All standards are prepared by spiking the appropriate volume of each standard solution into a glass soil impinger containing 10 ml of sparged, distilled/deionized water. All primary analytical standards should be stored in the volatile standards refrigerator when not in use (all volumes are given in μ 1).

Section No. 4.1.23

Revision No. 3

Date: March 17, 1992

Page 9 of 23

STANDARD ID #	1906	1907	1908	1909	1910
036	5.0	5.0	5.0	5.0	5.0
394	2.0	5.0	10.0	15.0	20.0
1301	1.0	2.5	5.0	7.5	10.0
1303	1.0	2.5	5.0	7.5	10.0
1307	1.0	2.5	5.0	7.5	10.0
1322	1.0	2.5	5.0	7.5	10.0
1354	1.0	2.5	5.0	7.5	10.0
CIS 1, 2 dichloroethene	1.0	2.5	5.0	7.5	10.0

GC/MS Standards

I.D. #7008 Tuning Compound

bromofluorobenzene

 $25\mu g/1$

I.D. #1001 Matrix Spiking Solution All compounds at 25 μ g/l

1,1-dichloroethene benzene trichloroethene toluene chlorobenzene

I.D. #1301 All Compounds at 100 ug/ml

methylene chloride	trich
1,1-dichloroethene	1,1-d
chloroform	carbo
1,2-dichloropropane	trich
1,1,2-trichloroethane	dibro
2-chloroethyl vinyl ether	tetra
chlorobenzene	trans
1,2-dichloroethane	1,1,1
bromodichloromethene	trans
benzene	cis-1
bromoform	1,1,2
toluene	ethyl

lorofluoromethane dichloroethane on tetrachloride loroethene omochloromethane chloroethene s-1,2-dichloroethene l-trichloroethane s-1,3-dichloropropene 1,3-dichloropropene 2,2-tetrachloroethane ylbenzene

Section No. 4.1.23 Revision No. 3 Date: March 17, 1992 Page 10 of 23

I.D. #1322A

Compounds @ 100 ug/ml

Compounds @ 50 ug/ml

acetone
methyl ethyl ketone
4-methyl-2-pentanone
2-hexanone
carbon disulfide
styrene
o-xylene

m-xylene p-xylene

I.D. #1322 All 1322A compounds @ 100 ug/ml

I.D. #1354

1,2-dibromo-3-chloropropene crotonaldehyde

Conc-200 ug/ml Conc-1000 ug/ml

All other compounds @ 100 ug/ml

cis-1,4-dichloro-2-butene trans-1,4-dichloro-2-butene ethylmethacrylate 1,1,1,2-tetrachloroethane 1,1,1-trichlorotrifluoroethene 1,2-dibromoethane dibromomethane 1,2,3-trichloropropane iodomethane

1,1,1-trichlorotrifluoroethene 3-chloropropene
1,1,2-trichlorotrifluoroethene

I.D. #1303 All compounds @ 1000 ug/ml

acrolein acrylonitrile

I.D. #1307 All compounds @ 100 ug/ml

chloromethane vinyl chloride

bromomethane chloroethane

Section No. 4.1.23 Revision No. 3 Date: March 17, 1992 Page 11 of 23

I.D. #036 Internal Standards

Conc-50 ug/ml

bromochloromethane p-difluorobenzene d5-chlorobenzene

> I.D. #394 Surrogates

Conc-50 ug/ml

d4-1,2-dichloroethane d8-toluene p-bromofluorobenzene

The surrogate recovery ranges are:

	Solid <u>OC Limit</u>
d ₄ -1,2-dichloroethane	70-121%
bromofluorobenzene	74-121%
d ₈ -toluene	81-117%

6.8 Standard Analysis

Immediately after preparation of standards, they are injected via a Teflon stopcock on the Tekmar into a purge vessel and purged for 11 minutes. Samples are acquired using the AC program following the sequence:

AC filename # number of scans to acquire (RETURN)

Enter the appropriate information for sample description, including instrument ID and operator ID (as prompted by the program). When information has been entered and the instrument reads "ready," the Tekmar is switched to the desorb position which introduces the sample onto the head of the column. Data acquisition will continue unsupervised through acquisition of the designated number of scans. Enough scans should be acquired to ensure complete elution of the final compound.

Section No. 4.1.23 Revision No. 3 Date: March 17, 1992 Page 12 of 23

6.9 Primary and Secondary Ions and Detection Limits

benzene bromodichloromethane bromoform bromomethane carbon tetrachloride chlorobenzene	78 83 173 94 117	85, 127 175, 254	(μ g/kg) 5
bromodichloromethane bromoform bromomethane carbon tetrachloride	78 83 173 94	85, 127 175, 254	5
bromodichloromethane bromoform bromomethane carbon tetrachloride	83 173 94	175, 254	
bromoform bromomethane carbon tetrachloride	173 94	175, 254	
bromomethane carbon tetrachloride	94	•	
carbon tetrachloride		06	10
	117	96	5
chlorobenzene		119	5
	112	77, 114	5
chloroethane	64	66	10
chloroform	83	85	5
chloromethane	50	52	10
1,2-dibromo-3-chloropropa	ne 75	155, 157	10
dibromochloromethane	129	127	5
1,2-dibromoethane	107	109, 188	5
dibromomethane	93	95, 174	10
1,1-dichloroethane	63	65, 83	5
1,2-dichloroethane	62	98	5
1,1-dichloroethene	96	61, 63	5
cis-1,2-dichloroethene	96	61, 98	5
trans-1,2-dichloroethene	96	61, 98	5
1,2-dichloropropane	63	112	5
ethylbenzene	· 91	106	5
methylene chloride	84	86, 49	10
styrene	104	78	5
1,1,1,2-tetrachloroethane		133, 119	5
crotonaldehyde	70	41, 39	100
ethylmethacrylate	69	41, 69	10
cis-1,4-dichloro-		•	
2-butene	88	53, 88,	75 15
trans-1,4-dichloro-	• •	33, 33,	
2-butene	53	75, 53,	89 15
iodomethane	142	127, 141	10
3-chloropropene	76	41, 39,	
1,1,1-trichloro-	. •	,,	
2,2,2-trifluoroethene	117	151,119,1	L53 10
1,1,2-trichloro-		101/11/	100 10
1,2,2-trifluoroethene	85	101,151,1	L03 10
1,1,2,2-tetrachloroethane		131, 85	10
tetrachloroethene	166	168, 129	5
toluene	92	91	5
1,1,1-trichloroethane	92 97		5
1,1,2-trichloroethane		99, 61 97, 85	5 5
trichloroethene	83 95	97, 85 130, 132	5 5

Section No. 4.1.23
Revision No. 3

Date: March 17, 1992

Page 13 of 23

	Analyte	Character	ristics	Detection
	•	Primary	Secondary	Limit
		Ion	Ion(s)	(µg/kg)
	trichlorofluoromethane	101	103	5
	1,2,3-trichloropropane	75	7 7	15
	vinyl chloride	62	64	10
	o-xylene	106	91	5
	m-xylene	106	91	5 5
	p-xylene	106	91	5
	acrolein	56	56, 55	90
	acrylonitrile	53	51, 52	120
	carbon disulfide	76	44	5
	2-hexanone	43	58, 41	15
	4-methyl-2-pentanone	43	58	15
	2-chloroethyl vinyl ether	63	43, 44	10
	cis-1,3-dichloropropene	75	39, 77	5
	trans-1,3-dichloropropene	75	39, 77	5
	acetone	43	58	10
Internal	Standards/Surrogates			
	4-bromofluorobenzene	95	174, 176	
	toluene-d ₈	98		
	1,4-difluorobenzene	114		
	chlorobenzene-d ₅	117		
	bromochloromethane	128	49, 130	
	d ₄ -1,2-dichloroethane	65	·	

6.10 Quantitation

Standards are quantitated using the RK program with option 2. This procedure allows individual compounds to be quantitated using an average fit to the response list of the most recently updated continuing calibration check standard. The RK procedure is initiated by typing the command in the sequence:

RK filename #2, linker (RETURN)

The linker for standards quantitation is WELL. All 59 compounds must be found for each standard in the five-point calibration. Compounds not found by the RK program can be found by the UPQUAN program in the sequence:

UFIND filename, linker -or-UPQUAN library ID # library entry (RETURN)

Section No. 4.1.23
Revision No. 3
Date: March 17, 1992
Page 14 of 23

After all compounds have been found, the quantitation list must be edited. This can be accomplished using the EQL program in the sequence:

EQL filename, filename (RETURN)

and deleting any blank entries on the quantitation list or multiple entries for the same compound from the UPQUAN program. Following the editing of the quantitation list, it must be sorted using the QSORT program in the sequence:

QSORT filename, linker (RETURN)

The linker remains WELL for standards quantitation. The quantitation list can then be reprinted by the MQ program in the sequence:

MQ filename; F2;H;E (RETURN)

which will print the compound list and F2 table. If a large number of compounds were not found by the RK program, the 11 table should be updated at this point so that compounds in subsequent samples will be located correctly. This can be accomplished by typing the following commands:

SET1 filename (RETURN)
SET2 linker (RETURN)
RKSL B'; E (RETURN)

To enter these commands, the computer must be in the Alternate Executive mode. If it is not in the Alternate Executive mode, typing DISSW will perform this task. After entering the above commands and updating the 11 table, type DISSW again to return to the normal operating mode.

6.11 Generating the Initial Calibration

After quantitation of all five standards, the initial calibration is generated using the EPAMP program in the sequence:

EPAMP linker (RETURN)

using the linker WELL. This program will then prompt the chemist to enter file-names of low, med-low, med, med-high, and high level standards. The EPAMP program will then generate a report detailing response factors for each standard, average response factors for all five standards, and percent relative standard deviations (RSD) for each

Section No. 4.1.23
Revision No. 3
Date: March 17, 1992
Page 15 of 23

compound. Missing response factors for one or more compounds will result in an incorrect initial calibration. Appropriate standards must be corrected using the above procedures followed by recreation of the initial calibration using EPAMP. Compounds designated as SPCC and CCC must also meet EPA initial calibration criteria (see SPCC and CCC criteria below). The initial calibration must also be inspected for any bad entries or unusual data points. This initial calibration is double checked and approved by either a Senior Operator or Data Review Specialist.

6.12 Definition of a Valid Initial Calibration

- One valid injection of each of five standard concentrations.
- All standard acquired under a valid tune.
- All compounds present in all five standards.
- All isomeric pairs must be resolved except (on packed columns) the 1,2-dichloroethene and o,p-xylene.
- Meets all CCC criteria.
- Initial calibration reviewed and signed off by Chemist III or Data Review Specialist.

6.13 SPCC Criteria

The following compounds must have an average response factor greater than or equal to 0.300 in the initial calibration.

- chloromethane
- 1,1-dichloroethane
- bromoform (0.250 for bromoform only)
- 1,1,2,2-tetrachloroethane
- chlorobenzene

6.14 CCC Criteria

The following compounds must have a percent RSD (between the five standards) of less than or equal to 30% in the initial calibration.

- vinyl chloride
- 1,1-dichloroethylene
- chloroform
- 1,2-dichloropropane
- toluene
- ethylbenzene

Section No. 4.1.23 Revision No. 3 Date: March 17, 1992 Page 16 of 23

6.15 Contents of the Initial Calibration Package

A complete initial calibration package must contain each of the following:

- BFB tuning and mass calibration forms
- Quantitation report form for each standard
- A labeled RIC for each of the five standards
- Initial calibration form generated by the EPAMP Program

7.0 Calibration Check Standard

7.1 Frequency

A calibration check standard should be run immediately after injection of BFB. If no valid WELL initial calibration exists on the instrument, a calibration check standard must be run after obtaining a valid initial calibration. Valid calibration check standard must be obtained before sample analysis.

7.2 Nominal Concentration

50 ug/kg
No substitution of standard concentration

7.3 Calibration Check Standard Preparation

The calibration check standard is prepared by injecting the following amounts of primary analytical standards into a 5-ml gastight syringe containing 5 ml of sparged, distilled/deionized water. All volumes are given in μ l.

1907	
5.0	
5.0	
2.5	
2.5	
2.5	
2.5	
2.5	
2.5	
	5.0 5.0 2.5 2.5 2.5 2.5 2.5

7.4 Standard Analysis

Analysis of the calibration check standard follows the procedure defined under an initial calibration.

Section No. 4.1.23 Revision No. 3 Date: March 17, 1992 Page 17 of 23

7.5 Quantitation

The calibration check standard is quantitated according to the procedure defined under an initial calibration.

7.6 Check Standard Calibration Form

Validity of the calibration check standard is checked using the EPAUP program, using the sequence:

EPAUP filename , linker (RETURN)

program sets library amounts correctly, updates the 11 table, and prints the continuing calibration form. This form lists percent differences in the shift standard response factors and initial calibration response factors. The calibration check standard must meet all SPCC and CCC requirements as established by the EPA. If not, creation of a new five-point calibration is necessary.

SPCC criteria and CCC criteria remain the same for specific compounds as in the initial calibration except the maximum percent RSD for the CCC compounds is 25 instead of 30.

7.7 Contents of the Standards Package

- BFB tuning and mass calibration forms
- A quantitation report form for the Check Standard
- Labeled RIC for the Calibration Check Standard
- Check Standard Calibration Form generated by EPAUP
- Quantitation report form for a valid blank
- Labeled RIC for the instrument blank
- Compound list for the instrument blank, which also provides surrogate information
- Internal Standard monitor for the blank
- Spectra of any hits in the blank
- Searches of any extraneous peaks in the blank RIC

8.0 <u>Instrument Blank</u>

8.1 Frequency

A valid instrument blank must be obtained to go with a valid BFB run and a valid check standard.

8.2 Instrument Blank Preparation

An instrument blank is prepared by filling a 5-ml gastight syringe with 5 ml of water. To this volume are added 5 μ l of Internal Standard #036 and 5 μ l of surrogate #394.

Section No. 4.1.23
Revision No. 3
Date: March 17, 1992
Page 18 of 23

8.3 Definition of a Valid Instrument Blank

A valid instrument blank must be obtained before the analysis of any samples. All compounds except for the common laboratory contaminants (defined in the USEPA Statement-of-Work as methylene chloride, acetone, toluene, and 2-butanone) must not be present at concentrations above the detection limit. Contractually, common laboratory contaminants may be present at up to five times the method detection limit. All other target compounds may not be present at levels greater than one-half the detection limit. CompuChem has found that under normal circumstances toluene and 2-butanone should not be present above the method detection limit. In addition, the CompuChem requirements for methylene chloride and acetone are as follows: If the first instrument blank contains methylene chloride and acetone at greater than twice the detection limit, notify a supervisor. Under special conditions the supervisor or his designee may allow concentrations of the common laboratory contaminants in the instrument blank up to the contractually allowed limits. Inside the laboratory, criteria for the four previously-mentioned compounds are used only when holding times are in jeopardy. Holding times can also be client specific.

8.4 Quantitation

Instrument blank quantitation is analyzed according to the procedure detailed under Sample Quantitation (section 9.5).

8.5 Generation of the Compound List

The instrument blank compound list is generated following the procedure under Sample Generation of the Compound List (section 9.6).

8.6 Generation of Spectra

Dual spectra and comparative spectra of any compounds found can be generated by typing

QLLGV (RETURN) (RETURN)

8.7 Library Searches

The instrument blank library searches are generated following the procedure under Sample Library Searches (section 9.8)

Section No. 4.1.23
Revision No. 3
Date: March 17, 1992
Page 19 of 23

9.0 Samples

9.1 Sample Storage and Holding Times

9.1.1 Procedures for Sample Storage

The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until analysis or extraction.

9.1.2 Regulatory Holding Times

VOA analysis of soil/sediment samples must be completed within 10 days of validated time of sample receipt (VTSR) for EPA CLP 2/88. For SW-846, Method 8240 the regulatory holding time is 14 days from the date of sampling.

9.2 Sample Preparation

Samples are provided to the laboratory in glass soil impingers containing 5.0 g of sample. They are temporarily stored in the VOA GC/MS refrigerator labeled #3 at 4°C ±2°C. Five ml of sparged, distilled/deionized water are added to the sample. To this volume of the sample are injected 5 ul of Internal Standard #036 and 5 μ l of Surrogate #394. Sample spikes and blank spikes also require the addition of 10 μ l of #1001, which contains 1,1-dichloroethylene, trichloroethylene, benzene, toluene and chlorobenzene.

9.3 Sample Analysis

Immediately after the sample is prepared, it is heated on a heating block at approximately 40°C and purged for 11 minutes. The sample is analyzed using the AC program in the form:

AC filename # number of scans to acquire (RETURN)

Under the subheading "SAMPLE" enter the appropriate information for sample description, including instrument and operator ID (as prompted by the program). When all information has been entered and the instrument reads ready, switch the Tekmar to the desorb preheat position and the sample will be introduced onto the head of the column. Data acquisition will continue unsupervised through acquisition of the designated number of scans. These procedures use subset linkers with compound lists that vary from the full list master linker. EPA solid uses ROCK1, Third Edition Solid + LS uses ROCK (the master linker).

Section No. 4.1.23 Revision No. 3 Date: March 17, 1992 Page 20 of 23

9.4 Quality Control

Quality Control (QC) sample types and frequencies are specified in CompuChem's Comprehensive QA Plan. Acceptance criteria, control limits, and corrective actions are also outlined in the QA Plan.

9.5 Quantitation

The sample is quantitated using the RK program with option 2. This procedure allows individual compounds to be quantitated using a calibration check standard for the particular analysis. The RK procedure is initiated by typing the command in the sequence:

RK filename #2, linker (RETURN)

using the linker appropriate for the type of analysis. If any internal standards, surrogates or other compounds are not found they can be found by using the UPQUAN program in the sequence:

UPQUAN LIBRARY ID # LIBRARY ENTRY (RETURN)

for each compound not found. If any internal standard must be UPQUANed then all compounds which reference that internal standard must also be UPQUANed to ensure that the amounts reported for those compounds will be accurate. If any compounds are UPQUANed, the quantitation list must be sorted to restructure compound entries. This can be accomplished by using the EQL program in the sequence:

EQL filename, filename (RETURN)

and deleting any duplicate entries as necessary. When the quantitation list has been edited, the quantitation list can be sorted using the QSORT program in the sequence:

QSORT filename , linker (RETURN)

using a linker appropriate for the type of analysis. The quantitation list can be reprinted using the MQ program in the sequence:

MQ filename ;F2;H;E (RETURN)

which will print a compound list and the F2 table.

Section No. 4.1.23
Revision No. 3
Date: March 17, 1992
Page 21 of 23

9.6 Generation of the Compound List

The compound list can be generated by using the CLISTE program in the form:

CLISTE filename, linker (RETURN)

with an appropriate linker for the type of analysis. This program will prompt the chemist to enter an appropriate compound list number and the weight of sample purged on the Tekmar.

9.7 Generation of Spectra

Dual spectra and comparative spectra of any compounds found can be generated by simply typing:

QLLGV (RETURN) (RETURN)

9.8 Library Searches

If any peaks exist in the RIC that do not correspond to entries in the compound list and are greater than or equal to 10% of the height of the closest internal standard, then a library search proves necessary to identify these compounds. This is accomplished by the UNKIDL program in the sequence:

UNKIDL filename , linker , # number of searches (RETURN)

using an appropriate linker and number of searched required by the analysis. This program will prompt the user for EPA #, the case #, the first scan of interest, the last scan of interest, and the correction factor (this can be found on the last page of the compound list). Library searches must be evaluated to see if any priority pollutants were found that are not present in the quantitation report. EPA methods require 10 searches, but 12 are produced in case a TCL is searched needlessly.

10.0 Evaluation of Data

For blanks, samples, sample spikes, and blank spikes, all surrogates must fall within the specified control limits. In addition, all internal standards must pass the criteria of the Internal Standard Area Monitor. Any samples that fail the above criteria must be reprepared and reinjected to obtain successful results. If multiple samples fail these criteria, the problem should be corrected before any further analysis of samples.

Section No. 4.1.23 Revision No. 3

Date: March 17, 1992

Page 22 of 23

Quantitated values for any compounds found in a diluted sample must also fall within the upper half of linear range for the multipoint (100 ug/kg - 200 ug/kg) or 1000 μ g/kg-2000 μ g/kg for acrolein, acrylontrile, and crotonaldehyde. Any sample with compounds outside this range that has not been analyzed using 5 ml must be diluted accordingly and reinjected for a successful result.

11.0 <u>Instrument Conditions</u>

11.1 Analytical Column

DB624 Megabore

Absorption Trap: OV1/TENAX/Silica gel

11.2 Gas Chromatographic

Actuation Time:

Carrier Gas: 30 ml/min Helium Flow Rate: Inj. Port Temp: 250°C GC Mode: Capillary Initial Temp: O°C Interface Temp: 250°C Initial Time: 2 min. Final Temp: 135°C Ramp Rate: 7°C/min. Final Hold Time: 0 min. Split Flow: N/A Sweep Flow N/A Split/Sweep

11.3 Interface

Type: Jet Separator Temp: 250°C

Temp: 250°C Solvent Divert Time: 0 min

11.4 Mass Spectrometer

Manifold Temp 80°C

Filament/Multiplier Actuation Time: 0 sec

Scan Speed: 0.7 sec/scan

First Mass: 35 Final Mass: 285

11.5 Tuning And Calibration

Tuning Compound: PFTB

Tuning Sensitivity: 100 Counts/ng on-column

Bromochloromethane

Calibration Compound: BFB Standard Identification #: 7008

Calibration Criteria: Attached Form VII
Calibration Frequency: Every 12 hours

Section No. 4.1.23 Revision No. 3

Date: March 17, 1992

Page 23 of 23

11.6 Analytical Standard

Identification #: 1906, 1907, 1908, 1909, 1910

Frequency of Analysis: Every 12 hours

11.7 Sample Identification

Preparation Code: XXX Vial Size: 40 ml screw cap Internal Standard ID Number: 036 Surrogate ID Number: 394

Label: White, CC# Storage: 4°C

11.8 Injection Procedure

Tekmar LSC2 or 0 I Corporation 4460A, 11 minute purge, 10 minute bake, frit sparger (medium porosity), 40-ml sparge flow. Nominally 5-ml sample volume.

11.9 Chromotographic Maintenance

General absence of peak tailing.

11.10 Miscellaneous

Quantitation Method: Library entry

Quantification Method Name: WELL

Worksheet: COMVSWS

Compound List Number: 382 Library Name(s): WE

File Naming Convention: XX012345YZZ

XX: Analytical Prefix

Y: Shift Indicator (A,B,C)

ZZ: Instrument Number

12345 Last five digits of CC#

11.11 Analytical Prefix Types

Calibration File: BF Standard: GS
Blank: GB Initial Sample Injection: GH
Sample Rejection: GR Sample Re-extraction: GR

11.12 Analysis Type

All samples require a search of all extraneous peaks greater than 10% of the closest internal standard, up to 10 searches.

Section No. 5.0 Revision No. 5 Date: June 20, 1991 Page 12 of 56

Table 5-2. Quality Control Information GC/MS Laboratory

ethod	Fraquency of Mathod Blanks	Acceptance Criteria	Corrective Action
24	once every 12 hours (Instrument Blank)	All surrogates within CLs (Attachment #1); All TCLs < 1/2 DL; All non-TCLs <25 % IS peak height	Decontaminate lines, trap; flush column, reanalyse until blank meets all criteria
325	> or = 1:20	All surrogates within CLs (Attachment #1); TCL phthalates < 2X DL; other TCLs < 1/2 DL; no more than 3 non-TCLs (excluding solvent by- products) >25% IS peak height	Halt analyses until problem identified and corrected; re-extract entire batch
8270, CLP Semivolatile SOW 2/88, ResWell Semivolatile	> or = 1:20	All surrogates within CLs (Attachment #1); TCL phthlates < 2% DL; other TCLs < 1 DL; no more than 3 non-TCLs/ non-solvents > 10% IS peak height	Malt analyses until problem identified and corrected; re-extract entire batch
8240 a CLP VOA Sow 2/88 Aqueous Natrix	once every 12 hours	All surrogates within CLs (Attachment #1); TCL common lab solvents < 2X DL; other TCLs < 1 DL; no more than 3 non-TCLs > 10% IS peak height	Decontaminate lines, trap; flush column, reanalyse until blank meets all criteria
8240, CLP VOA Sow 2/88 Solid Hatrix	> or = 1:20	All surrogates within CLs (Attachment #1); TCL common lab solvents < 5% DL; no more than 3 non-TCLs > 10% IS peak height	Halt analyses until problem identified and corrected; re-extract entire batch

Section No. 5.0 Revision No. 5 Date: June 20, 1991 Page 17 of 56

Table 5-2. Quality Control Samples GC/MS Laboratory

Nethod	Frequency of MS/MSD Pair*	Acceptance Criteria	Corrective Action
\$270, CLP Semivolatile SOW 2/88, and Reswell Semivolatile	> or = 1:20	Majority of recoveries and RPDs within control limits; (Attachments \$1, \$2, \$3)	Analyse LCS; if acceptable, report MS/MSD and LCS with data qualifier noting sample matrix interference(s)
8240, CLP VOA SOW 2/88 and Reswell VOA	> or = 1:20	Majority of recoveries and RPDs within control limits; (Attachments #1, #2, #3)	Analyse LCS; if acceptable, report MS/MSD and LCS with data qualifier noting sample matrix interference(s)

RPD - Relative Percent Difference * Natrix Spike/Matrix Spike Duplicate pair

Nethod	Frequency of Duplicates	Acceptance Criteria	Corrective Action
Direct Inject Wedified 624	> or = 1:20	TCL RPDs < or = 25% (TCL RPDs advisory*)	Halt analyses until problem is identified and corrected; reanalyse DUP to verify system control restored

RPD - Relative Percent Difference

TCL - Target Compound List

* Until statistical intralaboratory performance data are generated

"Pair" refers to the set of duplicate spikes

Section No. 5.0 Revision No. 5 Date: June 20, 1991 Page 18 of 56

Attachment #1
QC SURROGATE SPIKE RECOVERY CONTROL LIMITS
GC/MS LABORATORY

Method	Surrogate Spike Compound	Aqueous* (% Recovery)	Solid/Waster (% Recovery)
VOA	D4-1,2-Dichloroethene	76-114	70-121
VOA	4-Bromofluorobenzene	86-115	74-121
VOA	Da-Toluene	88-110	61-117
B N	D5-Nitrobensene	35~114	23-120
BN	D10-Pyrene	40-130++	17-125++
BN	D14-Terphenyl	33-141	18-137
BX	2-Fluorobiphenyl	43~116	30-115
Acid	2-Fluorophenol	21-100	25-121
Aold	2,4,6-Tribromophenol	10-123	19-122
Acid	D5-Phenol	10-94	24-113

^{*}Limits derived from EPA-CLP 2/88 Statement-of-Work. For non-CLP analyses, limits are subject to change based upon updated intralaboratory statistical performance data; all recoveries must be within control limits. For CLP analyses, one acid and one RN surrogate may fail control limits in a sample or MS/MSD, minimum 10% recovery required.

^{**}Laboratory optional surrogate only; no action limits at this time.

Section No. 5.0 Revision No. 5 Date: June 20, 1991 Page 19 of 56

Attachment #2 QC SPIKE RECOVERY CONTROL LIMITS GC/MS LABORATORY

Method	spike Compound	Aquecus* († Recovery)	Solid/Waster (% Recovery)
YOA	1,1-Dichlorothene	61-145	59-172
VOA	Trichloroethene	71-120	62-137
VOA	Chlorobensene	75-130	60-133
VOA	Toluene	76-125	59-139
VOA	Benzene	76-127	66-142
BN	1,2,4-Trichlorobenzene	39-98	38-107
BN	Agenaphthene	46-118	31-137
BN	2,4-Dinitrotoluene	24-96	28-89
BN	Pyrene	26-127	35-142
BN	N-Nitroso-Di-N-Propylamine	41-116	41-126
BN	1,4-Dichlorobensene	36-97	28-104
Acid	Pentachlorophenol	9-103	17-109
Acid	Phenol	12-89	26-90
Acid	2-Chlorophenol	27-123	25-102
Aoid	4-Chloro-3-Methylphenol	23-97	26-103
Acid	4-Witrophenol	10-80	11-114

^{*}Limits derived from EPA-CLP 2/88 Statement-of-Work; for non-CLP analyses, limits are subject to change based upon updated intralaboratory statistical perpormance data.

Section No. 5.0 Revision No. 5 Date: June 20, 1991 Page 20 of 56

Attachment #3
MATRIX SPIKE DUPLICATE RELATIVE PERCENT DIFFERENCE (RPD) LIMITS*
GC/MS LABORATORY

Method	Matrix Spike Compound	Aqueous* (RPD)	Solid/Waster (RPD)
VOA	1,1-Dichlorothene	14	22
VOA	Trichlorosthens	14	24
VOA	Chlorobensene	13	21
KOV	Toluene	13	21
VOA	Benzene	11	21 21 21
BN	1,2,4-Trichlorobensene	28	23
BN	Acenaphthene	31	19
BN	2,4-Dinitrotoluene		47
BN	Pyrene	3 8 31	36
BN	N-Nitroso-Di-N-Propylamine	38	38
24	1,4-Dichlorobensene	28	27
yeiq	Pentachlorophenol	50	47
Acid	Phenol	42	35
bioA	2-Chlorophenol	40	50
yord	4-Chloro-3-Methylphenol	42	33
hoid	4-Nitrophenol	50	50

^{*}Limits derived from EPA-CLP 2/88 Statement-of-Work; for non-CLP analyses, limits are subject to change based upon updated intralaboratory statistical performance data.

DELIVERABLES CODE FOR REPORT FORMAT

Style 9 includes:

- o Cover letter
- o Table of Contents
- o Chronicle (including QC summary)
- o Case Narrative
- o Method Reference
- o QA Notices (if applicable)
- o Chain-of-Custody (if received)
- o Compound List
- o RIC (sample)
- o Quant. Report
- o Spectra
- o Library Search Form IV (if applicable)
- o Library Search Spectra (if applicable)
- o Blank Compound List
- o RIC (blank)
- o Quant. Report
- o Spectra
- o Matrix Spike/Matrix Spike Duplicate
- o RIC (spikes)
- o Quant. Report
- O Tuning Summary (for sample, blank & spikes)
- o Calibrations (Initial & Continuing for sample blank & spikes)
- o RIC (standards: includes, in order, samples, blank & spike)
- Quant. Report (sample, blank & spike)

APPENDIX B

EMS HERITAGE LABORATORIES, INC.

STANDARD OPERATING PROCEDURE FOR CHROMIUM VI ANALYSIS AND OTHER INFORMATION

APPENDIX B

EMS HERITAGE LABORATORIES, INC.

STANDARD OPERATING PROCEDURE FOR CHROMIUM VI ANALYSIS AND OTHER INFORMATION

TABLE OF CONTENTS

B. 1	Additional Information on the Hexavalent Chromium Standard Operating Procedure
B.2	Hexavalent Chromium Standard Operating Procedure
B.3	Chain-of-Custody Procedures
B.4	Standard Operating Procedures for Data Package Assembly
B.5	Data Reduction, Validation, and Reporting
B.6	Performance and System Audits

APPENDIX B.1

EMS HERITAGE LABORATORIES, INC.

ADDITIONAL INFORMATION ON THE HEXAVALENT CHROMIUM STANDARD OPERATING PROCEDURE

ADDITIONAL INFORMATION ON THE HEXAVALENT CHROMIUM SOP

The following additional information applies to the attached SOP for Hexavalent Chromium analysis.

- 1. Sample matrix: water.
- 2. Sample size: 250 ml.
- 3. Preventative maintenance: the spectrophotometer is checked quarterly by checking the wavelength with a standard filter.
- 4. Calibration standards: the calibration standards are prepared from Fisher (or equivalent) K₂Cr₂O₇ crystals at the following concentrations: 0.005, 0.010, 0.025, 0.05, 0.1, 0.25, and 0.5 ppm. The standards are stored at room temperature for no more than 6 months.
- 5. Quality Control samples codes:

BLA01:

Blank (distilled water containing same reagents as the sample.)

LCS02:

Laboratory control sample (0.2 ppm, prepared from 1000 ppm Fisher

stock solution.)

DUP01:

Duplicate

SPIO1:

Spike (0.025 ppm, prepared from 1000 ppm Fisher stock solution.)

VER01:

Continuing calibration standard (0.025 ppm, prepared from 1000 ppm

Fisher stock solution.)

6. Data deliverables: the final data package will included the following items:

Table of contents
Case narrative
Initial and continuing calibration results
Raw data
Calculated results
QC sample results
Chain-of-custody records

7. Chain-of-custody procedures: see attached SOPs.

APPENDIX B.2

ERM HERITAGE LABORATORIES, INC.

HEXAVALENT CHROMIUM STANDARD OPERATING PROCEDURE

EMS Test Code: M310.6 Referenced Methods: APHA 312 A SW846 7196 Rev. #1

HEXAVALENT CHROMIUM

I. INTRODUCTION

Chromium may exist in two forms, trivalent and hexavalent. The hexavalent form is more commonly occurring, and it is also the form of major concern. Hexavalent Chromium has a corrosive effect on skin and may produce ulcers on prolonged contact. Hexavalent chromium is a known carcinogen. Chromium is used predominantly in industrial processes and may enter water supplies through waste discharge. Compounds containing chromium are also added to cooling water lines to help prevent corrosion.

A. Detection Limit

0.01 mg/l

B. Regulatory Limit

Discharge to waterways 0.1 mg/l (Illinois)

General standard for the waters of Illinois 0.05 mg/l

Pretreatment limit will vary according to industrial user category and POTW requirements

C. Holding Time

24 hours

D. Preservative

Cool, 4. C, not filtered for surface water, filtered (0.45 u) for subsurface water.

E. Special Considerations

Hexavalent Chromium has a holding time of only 24 hours so samples must be analyzed as soon as possible after sampling, as hexavalent chromium is reduced to the trivalent form over time.

II. APPARATUS AND REAGENTS

- A. Spectrophotometer, 540nm
- B. Plastic cups

Page 2 05 4

- C. 25 ml graduate cylinder
- D. 1+1 H₂SO₄

Preparation: Using a volume/volume dilution, dilute one part sulfuric acid to one part water. CAUTION:

Always add acid to water, never the reverse. This reagent should be prepared in a cold water bath, as intense heat is generated.

1. L - -

E. Powder pillows chrome Ver# (HACH Chemical Co.)

ė.

F. Stock Hexavalent Chromium - 50 mg/l (1 ml = 0.05 mg)

Preparation: Analytically weigh 141.4 mg K₂Cr₂O₇, dissolve and bring up to volume in a one liter volumetric flask. The concentration of this solution is 50 mg/l Hexavalent Chromium. Holding time 6 months. Use caution when handling this reagent it is a known carcinogen.

III. PROCEDURE

A. Theory

Hexavalent Chromium in this procedure i determined colorimetrically by reaction with diphenylcarbazide in an acidic solution. A redviolet dye is produced which can be measured spectrophotometrically at 540 nm.

B. Method

- 1. Turn on the spectrophotometer and set wavelength to 540 nm, and allow instrument to warm up for 15-20 minutes.
- 2. Use 25ml of sample and standards.
- 3. Add 2 3 drops 1+1 H_2SO_4 and swirl.
- 4.2 Add 1 powder pillow and swirl until powder has dissolved

(approximately 30 seconds).

5. Read absorbance.

IV. QUALITY CONTROL

Jung 2 5% 4

- A. A curve of at least 5 standards will be generated. Linear range is to 1 ppm.
- B. BLA01 once per run. Must be below detection limit reported.
- C. LCS02 1 per run, recovery must be $100\% \pm 10\%$.
- D. DUP01 1 per run or 10 samples. RPD must be less than 20% or 5x D.L. ± D.L.
- E. SPI01 1 per run or 20 samples, recovery must be $100\% \pm 20\%$.
- F. VER01 Every 10 samples, mid range recovery must be 100% ± 10%.

V. CALCULATIONS

Samples concentration is calculated from a linear regression curve of the standards. Multiply this value times any dilutions to get final sample concentration.

mg/l (off curve) x Dilution = mg/l Hexavalent Chromium

VI. TROUBLESHOOTING/INTERFERENCES

A. If the sample has any appreciable color or turbidity of it's own, it will rad a falsely high absorbance. This can be corrected by running a "color blank", which is the sample's absorbance without any reagents added. This value is subtracted from the sample absorbance to yield a corrected value.

Sample absorbance - Color Blank absorbance = Corrected Sample absorbance.

B. The diphenylcarbazide reaction is nearly specific for chromium, therefore chemical interferences are uncommon.

Hexavalent molybdenum and mercury will react to form a color in solution with diphenylcarbazide but the lower pH of the chromium analysis will keep the color intensity low and interference is unlikely. Iron concentrations greater than 1 mg/l will produce a yellow color but again the color is generally

not intense and therefore usually provides no difficulty in analysis. Vanadium can interfere strongly but concentrations up to 10 times that of chromium can be tolerated.

VII. **SAFETY**

Hexavalent Chromium is a known carcinogen.

1 + 1 H₂SO₄ is corrosive

Safe laboratory practices should be used at all times

VIII. CLEAN UP

The Hexavalent Chromium samples and standards may be disposed of down the sink when flushed thoroughly with water.

9002\jk10m

APPENDIX B.3 EMS HERITAGE LABORATORIES, INC. CHAIN-OF-CUSTODY PROCEDURES

AMPLE CUSTODIANS

Par 15 8

2.0 THE DUTIES AND RESPONSIBILITIES OF SAMPLE CUSTODIANS

Duties and responsibilities of the sample custodian shall include but not be limited to:

- 2.1 Receiving samples
- 2.2 Inspecting sample shipping containers for presence/absence and condition of :
 - 2.2.1 custody seals, locks, "evidence tape," etc.
 - 2.2.2 container breakage and /or container integrity
- 2.3 Recording condition of both shipping containers and sample containers (bottles, jars, cans, etc.) in appropriate logbooks or fields in the computer lab information management system (LIMS), and/or on appropriate client forms.
- 2.4 Signing appropriate documents shipped with samples (i.e. airbills, chain-of-custody record(s), etc.)
- 2.5 Verifying and recording agreement or non-agreement of information on sample documents (i.e., sample tags, chain-of-custody records, traffic reports, airbill, etc.) in appropriate logbooks or computer fields and/or on appropriate forms. If there is non-agreement in these documents, recording the problems, contacting the client for direction and informing the appropriate laboratory personnel. (The corrective action directions shall be documented in the project file.)
- 2.6 Initiating the paper work for sample analyses on appropriate laboratory documents and/or in the laboratory data system as required for initiation of the analysis sequence according to standard laboratory procedures.
- 2.7 Marking or labeling samples with laboratory sample numbers generated by the computer and cross referencing laboratory numbers to client numbers using the appropriate fields in the computer log-in procedure and/or chain-of-custody sheet.
- 2.8 Placing samples and spent samples into appropriate storage and/or secure area.
- 2.9 Controlling access to samples in storage and assuring that laboratory standard operating procedures are followed when samples are removed from and returned to storage.
- 2.10 Monitoring storage conditions for proper sample preservation such as refrigeration temperature and prevention of cross-contamination. The temperature of the sample storage areas recorded daily and any deviations from 4 ± 2 degrees C noted. Samples are opened (when necessary) under a fume hood individually to avoid cross contamination.

9/90 EMSIN GLP.01-9

THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT. Preservation of volatile vials with HCl will be done under the charcoal fume hood in the mass spec VOA prep lab (lab #11) and the date, lot number and other information entered into the logbook.

- 2.11 Returning shipping containers to the proper sampling teams.
- 2.12 Maintaining custody of the sample tracking logbook.

THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT.

BY _ Eym _ :ATE _ 12/5/9,

Page 2 of 8

3.0 STANDARD OPERATING PROCEDURES FOR SAMPLE RECEIVING

- 3.1 Upon receipt, examine the shipping container and record the following information in the computer LIMS system or on an appropriate form.
 - 3.1.1 Presence/absence of custody seal(s) on the shipping containers(s).
 - 3.1.2 Custody seal number(s) and condition (i.e. intact, broken, absent) when present.
- 3.2 Open the shipping container, remove the enclosed sample documents and record the presence/absence of the following forms and their respective numbers in the LIMS system or on an appropriate form:
 - 3.2.1 The chain-of-custody record(s).
 - 3.2.2 Record the project identification numbers.
- 3.3 Remove sample containers and record in the LIMS system or the appropriate form:
 - 3.3.1 Condition of samples (intact, broken, leaking, etc).
 - 3.3.2 Presence/absence of sample tags.
 - 3.3.2.1 Record sample tag document control numbers.
 - 3.3.2.2 Compare with chain-of-custody record(s).
 - 3.3.2.3 Document whether or not these numbers agree.
- 3.4 Compare the documents listed below to verify the information contained on them. Both agreement among the forms and any discrepancies found will be documented. If discrepancies are found, contact client or project manager for clarification (and/or resolution) and notify appropriate laboratory personnel. The documentation of corrective action instructions and the source of those instructions will be forwarded to the Project Manager for inclusion in the data file.
 - 3.4.1 Chain-of-custody records
 - 3.4.2 Sample tags
 - 3.4.3 Laboratory Task Order
- 3.5 If all samples recorded on the chain-of custody record were received by the lab and there are no problems observed with the sample shipment, the custodian will sign the chain-of-custody record in the "received for laboratory by:" box.

9/90 EMSIN GLP.01-11 THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT.

they be d

If problems are noted, sign the chain-of-custody record and then note problems in remarks box or reference other forms that describe the problems in detail.

- 3.6 The sample custodian should record the laboratory sample numbers assigned by the computer on the sample containers and tags.
- 3.7 Samples received when the sample custodian and other designated recipients are absent will be placed in the proper lab location. The person receiving the shipping container will sign for the container, place it in the secure, refrigerated location and record the time, date and name of individual receiving the container.

The sample custodian or their designee will log-in the samples on the next business day. The date on the receipt form will be the date the form was completed.

3.8 Broken samples should be disposed of appropriately and the client notified that re-sampling is necessary.

THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT.

84 Kgm 115 12/5/91

124 4 55 8

4.0 STANDARD OPERATING PROCEDURES FOR SAMPLE IDENTIFICATION

4.1 In order to maintain sample identity each sample received must have a unique sample identification number. The laboratory identification number is a sequential number assigned by the LIMS computer. The LIMS system allows for the cross-reference of laboratory sample number and client number along with date of sample receipt.

Jung 5 35 8

- 4.2 Sample containers (bottles and extract vials) will be clearly identified with the appropriate sample number.
 - 4.2.1 During the sample receiving process, the sample custodian or other appropriate personnel will assure that each sample container is identified with a unique sample ID number and that number is recorded in the sample receipt documentation. The laboratory ID number, assigned by the LIMS system, is written on the sample container label and cap.
 - 4.2.2 Extract vials will have the sample ID numbers and the appropriate fraction (\underline{A} cid, \underline{B} ase, \underline{P} est etc.) written on the vial. These numbers should be recorded on the sample tracking documentation (benchsheets).

con ex X

5.0 STANDARD OPERATING PROCEDURES FOR SAMPLE STORAGE

- 5.1 The samples will be removed from the shipping container and stored in their original containers unless damaged.
- 5.2 Damaged samples will be transferred to another bottle or disposed of in an appropriate manner and this transferral/disposal will be documented in a logbook along with the date and initials of the analyst.
- 5.3 Samples and extracts will be stored in a secure area, on the shelves designated for that analysis or project.
- 5.4 The secure areas include the walk-in refrigerator #16 for Semi-volatiles and PCB/Pest, and refrigerator #25 general inorganic and metal samples and the refrigerators in the Mass Spec (VOA samples) and GC (Pest extracts) rooms. These areas are locked after normal working hours. These storage areas are maintained at 4 ± 2 degrees C. The temperature is monitored and recorded daily, by the receiving personnel or group.
- 5.5 The storage area will be kept secure at all times. The sample custodian will control access to the storage area through the receiving area and the building security system controls access from other doors.
- 5.6 Whenever samples are removed from storage, this removal will be documented in the proper logbook (extraction, analysis). The date of extraction or analysis and the analyst's initials will be entered into the LIMS system to permit sample tracking throughout the analysis process.
- 5.7 Samples and extracts will be stored after completion of analysis in accordance with the contract or until instructed otherwise by the client. At this time, samples are stored for 30 days after reporting and extracts for 90 days.
- 5.8 Standards will not be stored with samples. The working standards for PCB's/Pests, Semi-volatiles and Volatiles are stored in refrigerators in each analytical lab.
- 5.9 In order to satisfy sample chain-of-custody and confidentiality requirements, the following standard operating procedures will be followed.
 - 5.9.1 Samples will be stored in a secure area.
 - 5.9.2 Access to the laboratory will be through a monitored area. Other outside-access doors to the laboratory will be kept locked and access limited to employees only.

5.9.3 Visitors will sign a visitors log and be escorted by a plaza employee while in the laboratory. No laboratory area will be entered unless a member of the laboratory staff is present.

dage + 5-8

- 5.9.4 Refrigerators, freezers and other sample storage areas will be securely maintained or locked.
- 5.9.5 Only the designated sample custodian and the supervisory personnel of each lab group will have keys to locked sample storage area(s).
- 5.9.6 Samples will remain in secure sample storage until removed for sample preparation or analysis.
- 5.9.7 All transfers of samples into and out of storage will be documented in the appropriate analysis logbook.
- 5.9.8 After a sample has been removed from storage by the analyst, the analyst is responsible for the custody of the sample. Each analyst must return the sample/extract to the storage area before the end of the working day or prior to the end of his/her shift.

loge i _ y

6.0 STANDARD OPERATING PROCEDURES FOR TRACKING SAMPLE ANALYSES

- 6.1 Both the preparation and the analysis of samples will be documented.
- 6.2 All analysts will use the proper bench sheet for the analysis.
- 6.3 All notebook pages, bench sheets, graphs, computer printouts, chromatograms, GC/MS spectra and other laboratory project related documents will contain the date, signature (initials) of the analyst and other pertinent information as stated in the method or contract.
- 6.4 Upon completion of analysis, data will be filed in the appropriate sample files.
- 6.5 Copies of QA/QC will be placed in the appropriate file.
- 6.6 Instrument logs will be maintained for each gas chromatograph-mass spectrometer (GC/MS), gas chromatograph (GC), inductively coupled plasma (ICP) unit, atomic absorption unit (AA), and wet chemistry instrumentation equipment (e.g. CN-distillation apparatus, UV/VIS, colorimeter, etc). This logbook will contain the order of analysis, the instrument parameters, maintenance performed (and why) etc.
- 6.7 Copies of the instrument logs will be placed in the appropriate files, as necessary.
- 6.8 All sample preparation information will be documented in the laboratory notebook or bench sheet. All sample analysis data will be documented using instrument logbooks, computer printouts, chromatograms, analyst's laboratory notebooks, and/or other laboratory documents. When sample preparation or analysis is finished, the completed documents should be initialed and placed in the appropriate sample and/or project files.
- 6.9 If samples are run in batches, which may include several projects, all original batch analysis results will be filed in one sample/project folder. Copies of the results will be placed in each of the other sample/project folders and there shall be a reference to the file that contains the original analytical results documentation. Original calibration and QA/QC data should be treated in the same manner as the analytical documentation.
- 6.10 If, for any reason the original data cannot be placed in a sample/project file, this shall be documented and the location of the original data noted on the file inventory (xerox copies of the data will be included in the file).
- 6.11 All notes, comments and calculations made on or added to, project related data/documents will be signed and dated by the author/analyst/reviewer.

APPENDIX B.4

EMS HERITAGE LABORATORIES, INC.

STANDARD OPERATING PROCEDURES FOR DATA PACKAGE ASSEMBLY

1-y 10% 2

7.0 STANDARD OPERATING PROCEDURES FOR DATA PACKAGE ASSEMBLY

- 7.1 A procedure for organization and assembly of all documents relating to each client will be implemented by the appropriate Project Manager or designated person.
- 7.2 This procedure will ensure that all documents are compiled in one location for submission, and are arranged in the correct order.

Prepare data file folders:

- 7.2.1 Using appropriate file folders, assign one folder to each project.
- 7.2.2 Place all documents pertaining to one project in the folder.
- 7.2.3 Documents should be arranged by document type within the folders, i.e., all ICP data, all hard-copy Mass spectra together and in order as described in the contract or method.
- 7.2.4 These document files will be filed in one location and stored in a secure area.
- 7.2.5 The kinds of documents that will be contained in project and sample files are described in Tables 1 and 2.
- 7.3 The system must include a document numbering and inventory procedure.
 - 7.3.1 Each document type (see TABLE 1 and 2) is inventoried and listed on the cover sheet of the report. The number of pages and any unique identifier is listed.
 - 7.3.2 All documents pertaining to each project including, but not limited to, the following will be numbered and inventoried. All document groups should be clipped together. A document inventory list can be used for extensive projects (TABLE 3).
 - 7.3.2.1 Custody (internal and chain-of-) records, weekly reports.
 - 7.3.2.2 Laboratory logbooks, personal logbooks, instrument logbooks, bench sheets.
 - 7.3.2.4 Laboratory data (sorted by sample, when possible), calibration and quality control results.
 - 7.3.2.5 Data summaries and reports.

TON U.S. 2012 JUNIOT

- 7.3.2.6 All other documents, forms, or records referencing the samples.
- 7.3.3 Preparation of a document inventory
 - 7.3.3.1 A document inventory list provides a record of <u>all</u> documents, and their corresponding document numbers, that are included in the completed file.
 - 7.3.3.2 A separate document inventory list (See Table 3) is prepared for each project.
 - 7.3.3.3 The laboratory will retain copies of the document inventory lists for files reported to the client.

TABLE 1

ORGANICS DOCUMENTS

File inventory

* Chain-of-custody

Organics Analysis Data Summaries

Bench sheets and worksheets

Copies of instrument logbook pages

Sample tracing documents

Sample receipt logbook pages

** Internal custody records

Hard copies of mass spectra and chromatograms

QA/QC package

DFTPP/BFB calibration spectra and worksheets

Quality control reports

Standards analysis report forms, worksheets, spectra and chromatograms

Duplicate, matrix, surrogate spike results

GC/MS computer library search worksheets and accompanying spectra Related correspondence and/or memos

All other related documents

Each file must contain these documents or a memo explaining their absence (one memo may cover several documents).

^{*} If received with sample shipment

^{**} If used to supplement sample tracking system

TABLE 2

INORGANICS DOCUMENTS

File inventory

* Chain-of-custody

Inorganics Analysis Data Summaries

Copies of analysts' notebook pages and/or bench sheets

Worksheets

ICAP and AA instrument logbook pages

Sample tracing documents

Sample receipt log pages

** Internal custody records

Copies of instrument printouts

QA/QC data reports

Duplicate, blank, etc. analyses result

Related correspondence and memos

All other related documents

Each file must contain the following documents or a memo explaining their absence (one memo may cover several documents).

^{*} If received with sample shipment.

^{**} If used to supplement sample tracking system.

TABLE 3

DOCUMENT INVENTORY (EXAMPLE) Client No. 0000

Document Control #	<u>Document Type</u>	# Pages
Client#-(Project)#-Se	erial#	
0000-X-0001	Project File Document Inventory Shee	t
0000-X-0002	Case (Project) Narrative	
0000-X-0003	*Chain-of-custody	
0000-X-0004	Benchsheets	
0000-X-0005	Instrument Logbook Pages	
0000-X-0006	Sample Receipt Logbook Page	
0000-X-0007	Internal Custody Records	
0000-X-0008	ICP Data for Sample ######	
0000-X-0009	Volatile Data for Sample #####	
0000-X-0010	Semi-volatile Data for Sample ######	-
0000-X-0011	GC Chromatograms for Sample ######	_
0000-X-0012	Cyanide Run Sheet for Samples	_
0000 n 0012	#####-#####	
0000-X-0013	Calibration Information	
0000-Y-0013	(for each instrument)	_
0000-X-0014	Quality Control Reports (Blanks)	
	Standards Spectra and Chromatograms	
0000-X-0015		-
0000-X-0016	Matrix Spike/Spike Duplicate data	
0000-X-0017	All other related documents	

* If received with sample shipment Note: Each file must contain the following documents or a memo explaining their absence (one memo may cover several documents).

0000-X-0001

APPENDIX B.5 EMS HERITAGE LABORATORIES DATA REDUCTION, VALIDATION, AND REPORTING

12.0 Data Reduction, Validation and Reporting

12.1 Introduction

The laboratory analyst holds the key to the successful completion of valid and documentable analytical testing. Analysts must be provided with proper training, equipment and supervision so that they know and follow proper analytical procedures. It is management's responsibility to make available at the bench level, this QA Plan and it is the analysts' responsibility to know and to follow its' requirements. In addition to the QA Plan, portinent EMS Heritage SOPs, EMS Heritage written methods and published (EPA, etc.) methods, the analysts must be fully aware and have easy access to any specific contract requirements and DQO's. Data validation will never result in acceptable data if the proper QC types are not analyzed as dictated by the specific project or contract.

12.2 Data Reduction

This section outlines how the raw data are reduced (calculated) into reportable values.

- 12.2.1 All lab analysts share some portion of the responsibility for maintaining documentation of the testing being performed. In addition to the various bound notebooks which are maintained documenting standard preparations, instrument maintenance, instrument performance, logbook of logbooks, etc., analysts must maintain some combination of bound notebooks and/or original raw data printouts with bench sheets indicating the analytical run contents, sequence and results. Some documentation requirements under various testing programs may make the use of loose bench sheets in lieu of bound sequentially numbered notebooks an unacceptable procedure, in which case bound notebooks will be utilized exclusively. Exclusive use of bound notebooks may necessitate the use of loose sheets for data entry of results and associated information into the LIMS system.
- 12.2.2 All analytical run notebooks must contain the following information at a minimum:
 - 1. Analyst identification
 - 2. Date of analysis

^{*} Laboratory Information Management System

Section 12.0 Revision 0 Nov. 1, 1991 Page 2 of 22 21

- 3. Instrument identification
- 4. Analytical method (Edition or Version) used and any deviations
- 5. Date of initial calibration
- 6. Computer file name if applicable
- Concentration and source identification of all standards and spikes
- 8. EMS sample I.D. number
- 9. Reference to prep performed
- 10. Any comments pertinent to the analysis or sample conditions (homogeneity)
- 11. Any dilutions, concentrations, sample manipulations (initial and final weights/volumes, etc.)

Calculation of results from raw data if not contained in the bound lab notebook, will be maintained and filed along with the original hard copy of instrument printouts.

- 12.2.3 When data entry of results into LIMS is required (direct data file transfer not available), it will be necessary to summarize the final results and all information needed for data entry. A combination of the analytical run notebook, the instrument printouts/chromatograms, calculation work sheets, prep bench sheets/notebooks and summary of final results will be sufficient to re-construct the sample analysis. This simple final result summary will include at a minimum:
 - 1. Analyst identification
 - 2. Date of analysis
 - 3. Instrument identification
 - 4. Analytical method used
 - 5. Computer file names

Section 12.0 Revision 0 Nov. 1, 1991 Page 3 of 22.21

- 6. EMS sample I.D. number
- 7. Comment to appear on the final report
- 8. Any dilutions or concentrations involved
- 9. Final results with proper units and detection limits
- 10. Reviewer initials and date of review
- 11. Data entry operator and date of data entry
- 12.2.4 All instrument printouts/chromatograms must contain the following at a minimum:
 - 1. Analysts' initials and date of review of hard copy
 - 2. Sample or standard identification (and cone.)
 - 3. Computer file numbers
 - 4. Dilutions of concentrations
 - 5. Date and time of injection or analysis
 - 6. Units (µg/L, mg/L, mg/kg, etc.)
 - 7. Identification of samples that are spiked or duplicated (in addition to computer assigned "Q" numbers).
 - 8. Peaks chosen for quantitation and their identification.
- 12.2.5 Some testing will generate only bench sheets when no instrument printout is possible or practical (pH, titrations, etc.) and no bound notebook is maintained. In those cases this bench sheet must contain all information required in the analytical run notebook previously discussed and the following at a minimum:
 - 1. Reviewer initials and date of review
 - 2. Data entry operator initials and date of entry
 - 3. Page numbers

Section 12.0 Revision 0 Nov. 1, 1991 Page 4 of 2221

- 4. Calculated final results, %R of spikes, RPD of duplicates, etc.
- 5. Any raw data used to arrive at the final result
- 6. Results of the calibration curve (linear regression, log transforms, etc.)
- 12.2.6 All calibration curves conforming to a linear equation will be fitted by the least squares linear regression technique. This procedure will also be used for calibration curves that require log transforms or similar transforms. The formula for calculation of the regression line is as follows:

Mpete:

Y = mX + b

Y = Point on the y axis

m = Slope of line

X - Point on the x axis

b = y intercept

The least squares estimate of b and m uses the following equations:

$$M = \frac{\sum XY - \frac{\sum X\sum Y}{n}}{\sum X^2 - \frac{(\sum X)^2}{n}}$$

Other scientifically sound procedures/data reduction techniques may be used as permitted by the Chief Chemist and the QA Officer.

12.2.7 When using the external standard calibration technique the average calibration factor (CF) can be used in place of a calibration curve (non-linear) when the %RSD is less than 20% over the working range and linearity through the origin can be assumed. The calibration factor (CF) is calculated as follows:

*For multi-response pesticides/PCBs, gasoline, diesel, etc., use the total area of all peaks used for quantitation.

The concentration of each analyte in the sample may be determined by calculating the amount of standard purged or injected, from the peak response, using the calibration curve or the calibration factor (CF) above. The concentration of a specific analyte is calculated as follows:

Aqueous samples:

Concentration (ug/L) =
$$[(A_z)(A)(V_t)(D)]/[(A_s)(V_t)(V_s)]$$

where:

- A_z = Response for the analyte in the sample, units may be in the area counts or peak height.
- A = Amount of standard injected or purged, ng.
- A_{x} = Response for the external standard, units same as for A_{x} .
- V_i = Volume of extract injected, μL . For purge-and-trap analysis, V_i is not applicable and therefore = 1.
- D = Dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made, D = 1, dimensionless.
- $V_t = Volume of total extract, \mu L.$ For purge-and-trap analysis, V_t is not applicable and therefore = 1.
- $V_s = Volume of sample extracted or purged, mL.$

Section 12.0 Revision 0 Nov. 1, 1991 Page 6 of 22 3

By inspection of the above formula one observes that the concentration in the extract is:

 $(A_x)(RF)$

where:

$$RF(ResponseFactor)^* = \frac{A}{A_s}$$

'RF is the inverse of CF.

Nonaqueous samples:

Concentration
$$(ng/g) = [(A_x)(A)(V_i)(D)]/[(A_x)(V_i)(W)]$$

where:

W = Weight of sample extracted or purged, g. The wet weight or dry weight may be used, depending upon the specific applications of the data.

 A_x , A_y , A_z ,

12.2.8 When using the internal standard technique (all GC/MS analyses) the average response factor (RF) can be used in place of a calibration curve (non-linear) when the %RSD is less than 20% over the working range and linearity through the origin can be assumed. The response factor (RF)* is calculated as follows:

$$RF = (A_{\mu}C_{\mu})/(A_{\mu}C_{\mu})$$

where:

 A_{i} = Response for the analyte to be measured.

A_{ii} = Response for the internal standard.

 C_{ia} = Concentration of the internal standard, $\mu g/L$.

 C_s = Concentration of the analyte to be measured, $\mu g/L$.

*Also known as "Relative Response Factor" or RRF

The concentration of a specific analyte is calculated as follows:

Aqueous samples:

Concentration
$$(\mu g/L) = [(A_r)(C_h)(D)/(A_h)(RF)(V_s)]$$

where:

- A_x = Response of the analyte being measured, units may be in area counts or peak height.
- C_{is} = Amount of internal standard added to extract or volume purged, ng.
- D = Dilution factor, if a dilution was made on the sample prior to analysis. If no dilution was made, D = 1, dimensionless.
- A_{is} = Response of the internal standard, units same as A_{x} .
- RF = Response factor for analyte.
- V_s = Volume of water extracted or purged, mL.

Nonaqueous samples:

Concentration
$$(\mu g/kg) = [(A_s)(C_{is})(D)]/[(A_{is})(RF)(W_s)]$$

where:

W₁ = Weight of sample extracted, g. Either a dry weight or wet weight may be used, depending upon the specific application of the data.

 A_i , C_{in} , D, A_{in} , and RF have the same definition as for aqueous samples.

The RF from the daily standard (CCV) analysis is used to calculate the concentration in the sample unless specific methods dictate otherwise. Section 12.0 Revision 0 Nov. 1, 1991 Page 8 of 2221

12.2.9 Manual data entry is the responsibility of the Data Entry Supervisor who will assure that data entry staff have knowledge of the system in order that complete analytical information is entered accurately, in the correct format and labeled consistently. Data entry staff will not perform any calculations; all data entry items will be summarized and submitted on standard forms which have been reviewed by the responsible group leader for acceptability, accuracy and completeness.

All analyses in the data system are based on the "analytical run" concept. All data (prep or analytical, is grouped in the computer, exactly in the order analyzed (as it also appears on a benchsheet). All QC is included in the analytical sequence.

Automated data transfer is the responsibility of each respective Group Leader.

12.3 Data Validation - During Collection

The principal criteria to be used to validate data integrity during collection will be the following:

- 1. Calibration Criteria (Linearity, RF etc.)
- 2. Internal Standard Areas (organics)
- 3. Calibration Blank Value (CAL01)
- 4. Method Prep Blank Value (BLA02)
- 5. Reagent Blank Value (BLA01)
- 6. Result of calibration verification standard used to verify calibration (ICV01).
- 7. Results of (matrix) spiked non QC samples (DPS01, SPI01, SPI02, DPS02).
- 8. Results of surrogate recoveries (organics)
- 9. Results of replicate measurements (DUP01, DUP02, DPS02).
- 10. Results of Continuing Calibration Verification standard (CCV)

Section 12.0 Revision 0 Nov. 1, 1991 Page 9 of 22.21

- 11. Results of Laboratory Fortified Blanks and Laboratory Control Samples or QC Check Samples (LCS, DLCS, ICV02)
- 12.3.1 These measurements are to be made by the analyst who, using specific acceptance criteria, will either proceed with analyses or take corrective action. All QC data must be reviewed by the Group Leader to insure that all QC types have been completed and meet acceptance criteria. Group Leaders must also:
 - 1. Check raw data entries and calculations.
 - 2. Check extraction logs and benchsheets.
 - 3. Check Calibration integrity.
 - 4. Check Instrument/analytical logbooks.

The Group Leaders are responsible for assuring that all bench sheets submitted for data entry are accurate and adequate to allow for the rapid and error free recording of data into LIMS.

- 12.3.2 Group Leaders and the Sample Custodian are also responsible for checking and assuring that all internal chain of custody requirements and documentation of such are met.
- 12.3.3 In summary, Group Leaders will review all bench sheets or release computer files to LIMS after review. In addition 20% of all raw data will be reviewed by the Group Leaders to insure that the method was in-control during the analytical run. Group Leaders are responsible for initialing and submitting data for data eatry upon completion of their review. This completes the first step in review of the data integrity and data validity.

12.4 Data Validation - Post Completion

Subsequent to data entry, the Quality Assurance Unit is responsible for comparing bench sheets with actual data entered into LIMS. Such review will occur for 2-5% of all data entered. This activity will be documented and retained on file.

Refer to Figure 12.1, <u>Data Review Protocol</u> for the procedures used at EMS Heritage for data review and validation. The QA Officer and/or the QA Unit must also:

Section 12.0 Revision 0 Nov. 1, 1991 Page 10 of 32.21

- 1. Confirm that the goals for precision and accuracy on duplicate and spiked compounds are sufficient to achieve the goal of 99% completeness.
- 2. Confirm that all analytical QC precision and accuracy (the measurement system, not sample (matrix) related) is 100% acceptable or that the run has been rejected and corrective action taken.
- 3. Confirm that all client specific or contract specific QC requirements have been met.
- 4. Examine at least 5% of the raw data (e.g. chromatograms, AAS recorder outputs, burn record on a periodic basis to verify adequacy of documentation, confirm peak shape and resolution, and to assure that the instrument system is sufficiently sensitive and responsive.

12.4.1 Internal and External Assessments

External assessment of the adherence to the QA/QC policy and procedures is the responsibility of the Quality Assurance Officer. Audit forms have been developed in the QAP and are used at all EMS Heritage Laboratories to evaluate their adherence to QA/QC Procedures. External assessments are also accomplished via third party audits.

Internal assessment of the compliance to company QA/QC policies and procedures is conducted by the Laboratory Director and the Quality Assurance Unit or their designee. Review of documentation, use of blind duplicates and standards and monthly reference materials shall be used to evaluate the performance of each laboratory. Refer to Section 14.0, <u>Performance and Systems Andits</u> for the details of internal and external assessments.

12.4.2 Quality Control Policy and Procedures

The EMS Heritage Laboratories, Inc. QAP is mandatory at all laboratories. Specific procedures are required and regular quality control audits are performed to assure compliance to company policy. Quarterly quality control reports are required from the QA Units submitted through the QA Officer. The overall quality of analysis shall be compiled and summarized by the QA Officer, who will report this summary to the President of EMS Heritage. Acceptable levels of

Section 12.0 Revision 0 Nov. 1, 1991 Page 11 of 22 21

performance are presented and monitored by the QA Officer.

12.5 Laboratory Quality Control Checks

The types and frequency of laboratory QC Checks are presented in Table 11.5, Analytical Run Requirements/Frequency. This table presents basically three categories of QC:

- 1. Instrument Calibration OC
- 2. Instrumental Method QC (tests w/no prep. or analyzed w/o prep)
- 3. Analytical samples QC processed through all steps (prepped QC).

Instrument Calibration criteria must be met before any analyses are allowed to proceed.

Internal QC checks not related to calibration are discussed below.

12.5.1 Preparation or Method Blanks

Blanks are identified at EMS Heritage as either BLA01 or BLA02 (calibration blanks are treated as a calibration data point, CAL01). Those methods that do not differentiate a prep from the analysis, i.e. purge and trap volatile analyses, are labeled as BLA01, which is equivalent to a BLA02 for tests that do have a separable prep and analysis. The following control limits apply for all method blanks (BLA02) and reagent blanks (BLA01):

If sample results are greater than 10 times the method blank value or the sample results are below the detectable level, the analysis is acceptable. Otherwise, the following criteria will apply.

1. Organic Analytes: Less than the Reporting Detection level (PQL)

Exceptions to the rule will be for common lab solvents not to exceed 5 times the PQL, including but not limited to the following:

1. Acetone

^{*} Only the page pertaining to Chromium VI analysis is included.

Section 12.0 Revisión 0 Nov. 1, 1991 Page 12 of 22 21

- 2. Methylene Chloride
- 3. Toluene
- 4. Methyl Ethyl Ketone (MEK)
- 5. Methyl Isobutyl Ketone (MIBK)
- 6. Freon(s)
- 7. Phthalates
- 2. Inorganic Analytes: Less than the Reporting Detection Level (PQL)

Exceptions to the rule will be for the following metals - not to exceed 3 times the PQL:

- 1. Iron
- 2. Nickel
- 3. Zinc
- 4. Copper
- 12.5.2 Any target compounds in the blank will be reported. Any sample containing a similar amount of the compound outside the acceptable range will be re-analyzed when the source of the contamination is identified and removed.

Target compounds found to be BDL (Below Detection Limit) in the blank will be entered into the data base at 0.4 times the MDL.

12.5.3 Matrix Spikes

Matrix spikes are included as a routine protocol according to the frequency given Table 11.5, Analytical Run Requirements/Frequency. Matrix spikes serve only to show that an individual sample does or does not exhibit matrix interferences using the prescribed method; they do not definitively demonstrate that a given analytical method is out of control. Other QC types will be utilized to demonstrate that an analytical method is in control (QC check sample, LCS, etc.).

- 12.5.3.1 There are basically two types of spikes utilized and they are as follows:
 - 1. Pre-digestion/extraction spikes
 - 2. Post digestion spikes

Pre-digestion/extraction spikes are performed for all applicable analyses at the required (Table 11.5) frequency. The goals for accuracy are listed in Table 5.1, QA Targets for Precision, Accuracy and Method Detection Limits.* Those control limits are determined from actual data entered into the QCIS when a sufficient number of observations (minimum of 7) are available to determine meaningful statistical limits.

Accuracy will be a function of the spiking level chosen. Many methods and testing programs specify the spiking levels to be used; EMS Heritage will use those spiking levels where specified. As a general rule, spiking will be performed at approximately 10 to 20 times the MDL unless other factors preclude that choice.

Several methods have established multi-laboratory performance based control limits for spike recovery of specific matrices (water and soil) through the USEPA. While Table 5.1 contains actual EMS Heritage developed control limits, the control limits used will be those determined by the USEPA for those equivalent analyses (assumes similar spiking levels used). Surrogate matrix spikes will be treated in the same manner for organic analyses. Refer to the specific methods for further explanation.

- 12.5.3.2 The spike (%R) recovery <u>OC Acceptance Criteria</u> for those organic methods providing specific limits are as found in Table 12.1, <u>Organic Spike Recovery Criteria</u>.**
- 12.5.3.3 The spike recovery (%R) OC Acceptance Criteria for Chromium VI analysis must be $100\% \pm 20\%$.

^{*} Only the page pertaining to Chromium VI analysis is included.

^{**} Not included since it does not pertain to Chromium VI analysis.

Section 12.0 Revision 0 Nov. 1, 1991 Page 14 of 22 A

Post-digestion spikes (SPI01) are primarily used in metals analyses and are also referred to as the Method of Standard Additions (MSA). Two versions of this MSA are referenced in SW-846, Third Edition. The full method of standard additions generates a 4 point curve from which final results can be calculated. Another version of MSA is the Single-Point MSA, which essentially is a sample and matrix specific spike recovery.

The spike recovery criteria for a post-algestion spike is as follows:

SP101 Criteria: 85-115% Recovery

12.5.4 Duplicate Matrix Spikes (DPS01, DPS02)

Duplicate matrix spikes measure both accuracy (see previous section for matrix spikes) and precision. As with matrix spikes, USEPA has established control limits for precision of specified methods for waters and soils. Those control limits are given as Relative Percent Difference (RPD) and will be used as control limits by EMS Heritage when applicable. When the methods do not specify RPD control limits, Table 5.1 of this QAP will be utilized when a sufficient number of observations allow for usable statistical limits. A maximum of 50 percent RPD will be allowed for organic testing and 20 percent RPD for inorganic testing. RPD's exceeding these limits will be considered as out-of-control for Duplicate Matrix Spike analyses.

Section 12.0 Revision 0 Nov. 1, 1991 Page 15 of 22.21

Maximum Acceptable RPD RPD Inorganic Criteria: 0-20 RPD Organic Criteria: 0-50

12.5.4.1 The Precision Control Limits for those organic methods providing specific limits are given in Table 12.3, Organic Precision Control Limits.*

12.5.5 Reagent Water or Reagent Matrix Spikes (LCS. DLCS)

Reagent water or reagent matrix spikes will be used as additional QC checks to monitor the effectiveness of the method. These QC types will utilize either reagent water spiked w/analyte (also known as Laboratory Portified Blank) or a blank matrix (water, soil, sand, oil) which is known to exhibit minimal or no matrix interferences. EMS Heritage uses the acronyms of LCS (Laboratory Control Sample), DLCS (Duplicate Laboratory Control Sample) and/or ICV02 (Initial Calibration Verification - Prepped Standard) to identify the variations of these check samples. Analysis of this QC type will demonstrate that the method is in control when the Matrix Spike/Matrix Spike Duplicate (MS/MSD) recoveries are out-of-control. These QC types must be extracted/digested (if applicable) in the same preparation batch as the samples failing to meet QC criteria. Analysis may be optional for some organic tests when QC criteria for MS/MSD samples are met.

12.5.5.1 The reagent water or reagent matrix spikes (Laboratory Control Sample) are entered into the QCTS in LIMS as are all QC types. In addition, these QC samples are kept on control charts at the bench level. The minimum QC criteria to be utilized for these QC types will be the same criteria as for matrix specific precision and accuracy. More stringent criteria will be utilized where available. As a general rule the following criteria will apply for the Laboratory Control sample:

(LSC, DLCS, ICVO2): 80-120% Recovery (Accuracy)
DLCS: 20% RPD

For Chromium VI analysis, the recovery must be $100\% \pm 10\%$.

^{*} Not included since it does not pertain to Chromium VI analysis.

Section 12.0 Revision 0 Nov. 1, 1991 Page 16 of 22.4

12.5.6 Quality Control Check Samples - Performance Evaluation Samples (P.B. Samples)

Acceptance criteria for P.E. samples are set by the organization running the study (EPA, commercial vendors). All results of P.E. samples are summarized to management.

12.5.7 Quality Control Check Standards or Initial and Continuing Calibration Check Standards (ICV01, CCV)

For Chromium VI analysis, the check standard (VER01) must have a mid-range recovery of $100\% \pm 10\%$.

12.5.8 Duplicate Samples (DUP01, DUP02)

Control limits for duplicate samples are identical to those set for duplicate matrix spike samples

with one exception. Since duplicate precision is measured from analyte already present in the sample (no spike added) the level may not be detectable or may be very close to the detection limit. Concentrations of analyte less than 10 times the reporting detection limit (MDL, PQL, etc.) present in non-spiked (duplicate) samples will not be considered as out-of-control based on the QCTS data, but will be allowed a maximum RPD of 20 percent for inorganic analyses and 50 percent for organic analyses.

12.5.9 Methods Utilizing Internal Standards

The criteria which must be met for internal standard areas (when used) are found in Table 12.4, Internal Standard Area Criteria.

12.5.10Surrogate Recovery Criteria

Control limit criteria for methods which do not specify specific control limits will be developed from in-house data. EMS Heritage will not adopt criteria less stringent than the published method criteria. "Advisory" criteria will not require corrective action. Refer to Table 12.5, Surrogate Recovery Criteria for method specific control criteria.

12.6 Review of Project Data

Project Managers are responsible for review of the overall project data before submission to the client. All laboratory "Certificates of Analysis" (Reports)

* Not included since it does not pertain to Chromium VI analysis.

and QA Summaries are reviewed and approved only by the QA Unit (see Reporting) prior to delivery to the Project Manager.

Project Managers are responsible for the following review prior to releasing the data report package to clients:

- 1. Review of supporting documentation including, but not limited to:
 - a. Chain of Custody.
 - b. Laboratory Analysis Request or Task Order Forms.
- 2. Review of all contract or project deliverables for completeness and accuracy including but not limited to:
 - a. Reporting forms.
 - b. Selected Raw data including chromatograms/printouts.
 - c. Adherence to specific DQO's.
 - d. Analysis of all required QC Types at the required frequency.
- 3. Review of data for any obvious anomalous values, specifically those areas requiring knowledge of special project conditions possibly unknown to the QA Unit during review. Review large sets of data for comparability from one analytical batch or case to another in the project (QA Unit does not generally review an entire case at one time).
- 4. Adherence to reporting deadlines and shipment/delivery of report packages by the best means available to meet those deadlines.

12.7 Data Reporting

The Data Entry Supervisor is responsible for supervision of all data entry operators. The Data Entry (D.E.) Supervisor (or designee other than the original data entry operator) will verify 5 percent of all data entered into

Section 12.0 Revision 0 Nov. 1, 1991 Page 18 of 22 H

LIMS. Locations having only one D.E. person will not check data in this manner; this responsibility will fall solely upon the QA Unit of that location.

Validation of data entry is performed for all data at a 2-5 percent frequency and is the responsibility of the QA Unit. The QA Unit may delegate this task to the QA Assistant or other staff person trained to perform that function.

The QA Unit is responsible for reviewing all final reports prior to release to clients. Reports are to be reviewed for:

- 1. Completeness All parameters, detection limits, units, dates, descriptions, etc. must be complete and correct.
- Consistency All parameters must be internally consistent (CrVI ≤ Cr Total; TKN ≥ NH₃ - N, TS ≥ TDS, etc.)
- 3. Comparability All parameters and units must be reported in such a way that data sets can be evaluated relative to each other as necessary.
- 12.7.1 All modifications to samples in the LIMS system must be documented on the Sample Modification Form. Figure 12.2. Original entries or additions (comments, etc.) to original entries will not utilize this form unless the sample is a "status 8" (released) category. All modifications to test codes must be approved by the Project Manager or the QA Unit. Modifications to samples that are completed and released (LIMS status 8) are possible only by the QA Unit staff. Any report that has been sent to a client and subsequently modified in any way by the QA Unit must be identified as an "amended report" and the date of amendment and the initials of the person making the amendment will be given on the certificate of analysis.
- 12.7.2 Samples of final reports and computer (LIMS) QC summaries are contained in the attached Final Report Examples.

12.7.3 Special Reporting Requirements

Sample analysis failing QC criteria due to matrix related interferences or samples requiring dilutions, etc. may have been analyzed 2 or more times. EMS Heritage will report a maximum of two analyses in those cases, or each analysis if client desires.

Section 12.0 Revision 0 Nov. 1, 1991 Page 19 of 22.21

If a re-analysis of the sample fails the same criteria a second time and all other instrument criteria are in control, a matrix interference is assumed and both analyses may be reported. If only the reanalysis is in control, only the reanalysis will be reported.

Exceptions to this policy will be made for contract requirements and/or different client DQO's.

12.8 Data Storage

The following data is maintained for long term storage.

- bench sheets
- strip charts
- chromatograms, print-outs
- magnetic media used to store results
- invoices and financial statements
- project files
- customer data
- various raw data
- lab related periodicals
- 12.8.1 The records, hard copy and magnetic media, are kept on site for a period of six months to a year. As necessary, records may be transferred to an off-site records storage facility. The records storage facility must provide secure, access controlled and environmentally controlled storage of records. For all GLP (40 CFR, part 160) projects, records will be kept on-site.

After the completion of the requested work, records of the raw data, quality assurance data and reports will be kept by EMS for at least five (5) years unless otherwise required by law or regulations. Magnetic tapes are maintained, however, magnetic tapes have a storage life expectancy of less than ten (10) years.

Section 12.0 Revision 0 Nov. 1, 1991 Page 20 of 22 21

12.8.2 The following is an overview of how records are stored and retrieved at EMS Heritage.

12.8.2.1 Storing Records

To send a box off site to a records storage facility the following three steps will be followed.

- 1. A box is filled with material of a related nature. The box is then marked to show the contents and the related service group and destruction date.
- 2. The box is then taken to the off-site storage coordinator where the box is given a unique number. The unique number, the contents, the associated service group, and the date stored are recorded in a ledger. Once the box is entered into the ledger it is scheduled to be picked up or delivered to the off-site records storage facility.
- 3. The off-site records storage facility stores the box and gives it a unique location code. Once the location code is assigned off-site records storage facility contacts the off-site storage coordinator, who enters the location code into the box's ledger entry. This allows for the recall of individual boxes.

12.8.2.2 Recalling Records

To recall records from off-site storage requires the EMS box number and the records storage facility location code, both of which are kept in a records storage ledger. A copy of the ledger is kept by the off-site storage coordinator, who can also recall the box from storage.

12.8.3 The LIMS system located in Indianapolis is backed up on a three cycle program. Every week a backup of the system is done. Weekly backup tape sets are rotated every week. So, if the computer is backed-up with tapes set "A" this week tape set "B" is used the next week. The tape sets are rotated back and forth every other week, except the last week of the month. For the last week of the month tape set "C" is used. By using this method it is possible to restore the

Section 12.0 Revision 0 Nov. 1, 1991 Page 21 of 22 2

computer from as far back as one month. The tape sets are held at off-site storage, except when called back to do the backup. EMS Heritage also maintains over a month of "redo's" on tape that are backed up each day. "Redo's" are copies of the transactions needed to apply to an old backup of the database to bring it up to date. EMS Heritage also maintains a two week rotation of backups for the software. The software is backed up each day as well.

12.8.4 Data is not stored or organized per specific project unless contract or other client requirements dictate such. If storage by project is required, all raw data and final reports are copied and stored in a "central project file".

All original raw data and benchsheets are organized and stored in folders chronologically according to analytical batch and date of analysis.

######################################	19 56 - 127 56 0 - 1 19 56 - 120 3 0 - 1 19 56 - 130 56 0 - 2 10 59 - 130 56 0 - 2 10 59 - 130 56 0 - 1 10 59 - 130 56 0 - 1 11 65 - 65 12 52 - 121 34 56 0 - 1 13 52 - 121 1 0 - 1 14 76 - 138 34 0 - 1 15 56 - 65	
Graundwater Graundwater O11 3/8/3 NOO-Spac Water 8/8/8 8/8/8 8/8/8 Non-Spec Water O11 8/8/8	76 - 127	
6rgundwater 011 3/8/3 NOD-Spac Water 8/8/3 8/5/3 8/5/3 NON-Spec Water 011 8/4/8	74 - 129	2
9/8/3 Non-Spac Water 8/8/8 8/8/8 Non-Spec Water 011 8/8/8	130 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	
3/8/3 Non-Spac Water 3/8/8 8/8/3 Non-Spec Water 011 8/8/8 Non-Spec Water	130 56 0 - 130 56 0 -	
8/8/8 8/5/3 8/5/3 8/5/4 Mon-Spec Vator 011 8/4/5	1 1 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	
8/8/8 8/8/8 Non-Spec Vater 011 8/8/8 Non-Spec Vater	134 58 0 - 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	
8/8/8 Non-Spec Vater 011 8/8/8 Non-Spec Mater	1 1 0 0 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9
8/8/8 Non-Spec Vater 011 8/8/8 Non-Spec Mater	- 655 - 181 0 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 -	9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9
Non-Spec Vator O11 8/8/8 Non-Spec Mater	- 121 - 121 - 139 - 6 - 1	9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9
8/8/8 8/4/8 Non-Spec Mater	- 1E1 - 0 - 118 - 14 0 - 1	98 4
Non-Spec Mater	74 0 -	18
Non-Epec Meter	- 0 -	-
į		
	N - 0 - 141 - 18 TB	2
CADHIUM	6 56 - 114	
8/8/8	5 45 - 136 7 8 - 1	11
SECAL-7190 Non-Spec Later CERONICE	1 - 4 - 111 - 20 8	11
D11 CHRONIUM	3 04 - 136 1 4 -	a
	18 63 - 160 S7 6 - R	1
SECA-1-191 GT-01-01-01-01-01-01-01-01-01-01-01-01-01-	481 - LL 2	
SWESS-T196 NOT-SPEC HASEC HEXANGLENT CHROSIUM	1800XVM	
Grounduator MEXAVALENT	CHRONIUM R 87 - 113	
SESSESSION NON-SPEC Estal COPPER	1 98 - 98	
Graundwater	1 . 66 - 15	

Relative Percent Difference (Retrepi-rep2)/irepi+rep2)+100)

(GROZ. sq1)

Section 11.0 Revision 0 Nov. 1, 1991 Page 2 of 9

Table 11.5 ANALYTICAL RUN REQUIREMENT/FREQUENCY

					Individual	M QC						7	oc:	1.001 3 000 7.00 93 00000		* * * * * * * * * * * * * * * * * * * *
	Melbod Reference	sevor	BLA61	ccy*	C2DL04	DP501*	Demos,	SPIOI (G MSA)	B1.A02	(CLP) BLACC SPICT	er 105	(CLP) LCS/ SP801	SPI02 ¹	DPS02	DLÆ92	DLCS
Dimolved Solids	EPA	R, 10%	R. 5%	NA	NA	R, 10%	R. 10%	R, 10%	NA	NA	R, 5%	NA	NA	NA	5%	٠
ISE Fluoride	EPA	С	B,E 10%	B,E 10%	В	P, 5%	P, 5%	P. 5%	P. 5%	·	P, 5%	•	P, 5%	P, 5%	P, 5%	-
Formaldebyde	AOAC						R, 10%				, 5%					
Gross Alpha, Beta (Radioactivity)	EPA	NA	B,E 20%	B	•	•	R, 10%	•	N/A	NA	•		NA	NA	NA	-
Titrimetric Hardaess	ЕРА	R	B,E 10%	NA	-	•	R, 10%	. •	NA	NA	R, 5%	•	NA	NA	NA	-
Henryalent Chronium	SM	R	B,E 10%	B,E 10%	В	R, 10%	R, 10%	R, 10%	P, 10%	-	R, 5%	•	R, 10%	R, 10%	R, 10%	
Hexavalent Cr	sw	Ŗ	B,E 10%	B,E 10%	Ð	R, 10%	R, 10%	R, 10%	P, 10%	-	R, 5%	•	R, 10%	R, 10%	R, 10%	-
lodine	SM	С	B,E 10%	B.E 10%	-	R, 10%	R, 10%	R. 10%	NA	NA	R, 5%	-	NA	NA	NA	-
TKN	EPA	С	B,E 10%	B,E 10%		R, 10%	R, 10%	R, 10%	NA	NA	R, 5%		NA	NA	NA	
Nitrate-N (Brucine)	EPA	С	B,E 10%	B,E 10%		R, 10%	R, 10%	R, 10%	NA	NA	R. 5%	-	NA.	NA	NA	-
Nitrate-N (Colorimetric)	EPA	С	B,E 10%	B,E 10%	-	R, 10%	R, 10%	R, 10%	NA	NA	R, 5%		NA	NA	NA	
Nitrite-N	EPA	С	B.E 10%	B,E 10%		R, 10%	R, 10%	R, 10%	NA	NA	R, 5%	-	NA	NA	NA	
Nitrate-Nitrite-N	EPA	С	B.E 10%	B.E 10%		R, 10%	R, 10%	R, 10%	NA	NA	R, 5%		NA	NA	NA	·
Nuisance Dust	нгоги	R	R	B,E 10%	NA	NA	R, 10%	NA.	NA.	NA	NA	NA	NA	NA	NA	-
Oil & Greass	EPA413.1	R	R	NA			R, 10%	:	NA	NA			NA	NA	NA	-
Oil & Greage	SW 9071	R	R	NA.			R. 10%	_	NA	NA	R, 5%		NA.	NA	NA	

Section 12.0 evis: 0 November 1, 1991 Page 1 of 1

FIGURE 12.1 EMS LABORATORIES

DATA REVIEW PROTOCOL

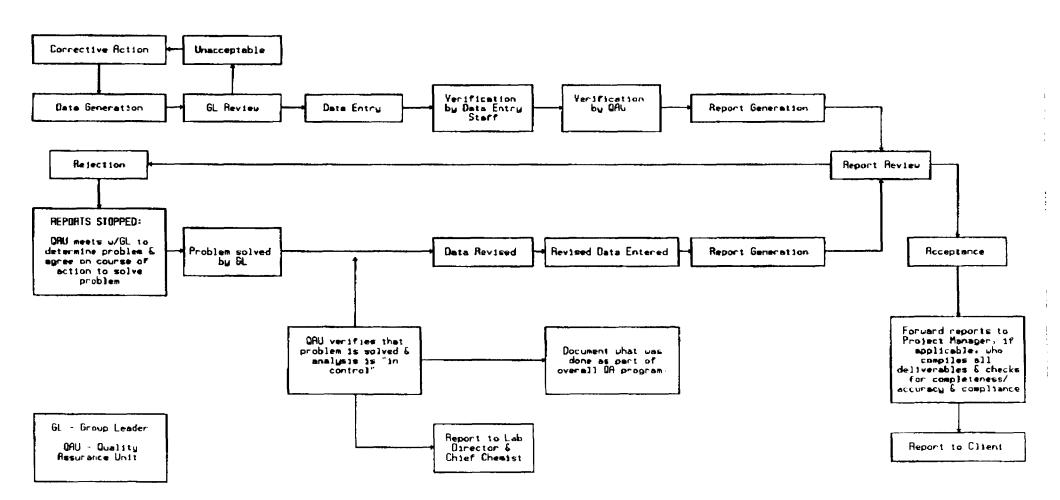


FIGURE 12.2

SAMPLE MODIFICATION FORM

Section 12.0 Revision 0 Nov. 1, 1991 Page 1 of 1

REQUEST FROM:	DATE
SAMPLE STATUS:	
SAMPLE NUMBERS:	
A STATE OF THE PARTY OF THE PAR	
CLIENT:	
TEST CODE ADDED/CANCELLED:	
TEST CODE CHANGED FROM	то
RESULTS MODIFICATIONS:	
Accompanies from the second of	
REASON FOR ADDITION OR CHANGE:_	
MODIFIED BY:	
PROJECT MANAGER'S APPROVAL INITI	
ACCOUNT MANAGER'S APPROVAL INFI	
•	
OLD PRICE:	NEW PRICE:

FINAL REPORT EXAMPLES

CERTIFICATE OF ANALYSIS

Service Location	Received	1 11 11
EMS HERITAGE LABORATORIES, INC.	23-APR-	
- 7901 W. MORRIS ST. INDIANAPOLIS, IN 46231	Complete	
(317)243-8305	05-JUN-1	91 90607684-59A \$ampled
(0-1/210 0000	13-JUN-	•
	10 0011	
Report To	B	il To
Sample D	escription	
·	·	
EAA OR TOR ACTO DIRECTION OF ADVENUE CAMPLES	CHOAC 2005	
FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES Analysis Date: 30-APR		Test::P130.4. 0
Parameter	Result	Det. Limit Units
INITIAL WEIGHT OR VOLUME	50	mL
TINAL WEIGHT OR VOLUME	50	mL
BARIUM ICP SW846-6010 Analyst: M. JAD Analyst: M. JAD Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS S	-91 Instrument: ICP	Tests M104.3. 0
Parameter	Result	Det. Limit Units
BARIUM	0.049	0.010 mg/L
CADMIUM ICP SW846-6010		of the other state of the
Analyst: H. JAO: Analysts: Date: 01-MAY Prep: FAA OR ICP: ACID DIGESTION OF AQUEOUS: S		Test: N10843. 0
Parameter CADMIUM	Result	Det. Limit Units
CADMIDIN	BOL	0.0050 mg/L
CHROMIUM ICP SW846-6010		
Analysti-M. JAO Analysis Date: 01-MAY		Test: M110.3. 0
Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS S	AMPLES SW846-3005	
Parameter	Result	Det. Limit Units
HROMIUM	BDL	0.010 mg/L
Mayer Ton Black Adda		
VICKEL ICP SW846-6010 Analysis Date: 01-MAY	-04 - 4 TOD	Test: M122.3. 0
Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS S	AMPLES SW846-3005	TERRIA DE LA CARRESTA DEL CARRESTA DE LA CARRESTA DEL CARRESTA DE LA CARRESTA DEL CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DEL CARRESTA DE LA CARRESTA DE LA CARRESTA DEL CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DEL CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRESTA DE LA CARRE
Peremeter	Result	Det. Limit Units
ICKEL	BDL	0.010 mg/L
SILVER ICP SW846-6010		
melysti M. JAC ACID DIGERTION OF ACHDING	-91 Instrument: ICP	Test: M130.3. 0
Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS S	AMPLES SW846-3005	
Paremeter	Result	Det. Limit Units
CILVER	BDL	0.010 mg/L

QUALITY ASSURANCE REPORT

Service Location Received Lab 10 EMS HERITAGE LABORATORIES, INC. 23-APR-91 A228370 7901 W. MORRIS ST. INDIANAPOLIS, IN 46231 (317)243-8305 PO Number Complete 90607684-59A 05-JUN-91 Sampled Printed 13-JUN-91

Sample Description

levick	et : M. JAO Per: S. ENDI D: FAA C		Review Date: 02-MA	Anatysis Date: 01-MAY-91 Instrument: ICP Review Date: 02-MAY-91 File ID: TION OF AQUEOUS SAMPLES SW846-3005			Test: M104.3.0 Run: R119838				
C Type	Identifier	Source	Parameter	True/Sampl	Spike Val	Observed	Units	% Rec	RPD		
נסער־	0239253	WP1083	BARIUM	2.00		2.06	mg/L	103	í		
502	2231974	A228372	BARTUH	0	2.00	1.92	mg/L	. 96.	4		
SP102	2231973	A228372	BARTUM	٥	2.00	1.84	mg/L	92	1		
במע	2239277	INORGANIC	BARIUM	5.00	1	4.52	mg/L	90			
101	c239276	NA	BARTUM			< 0.004	mg/L				
-દેર્ગ	0231971	NA	BARIUM	20.0		18.9	mg/L	94	1		
BLAOZ	9231972	NA	BARIUN			< 0.004	mg/L				
PLE	A228370		See Certificate of Analysis								
,	9239275	INORGANIC	BARIUM	5.00		4.81	mg/L	96			
BLA01	9239274	NA.	BARTUM	· · . · ·		< 0.004	mg/L				

Revie	st : M. JAO wer: Ş. END	ERSEN	Anatysis Review	Date: 01-MAY-91 Instrum Date: 02-MAY-91 File ID AQUEOUS SAMPLES SW			Test: M108:3:1 Run: R119838		
← Type	Identifier	Source	Parameter	True/Sampl	Spike Val	Observed	Units	% Rec	RPD
10701	02 392 54	WP1083	CADMIUN	0.400	,	0.425	mg/L	106	
1 01	0239259	WP1083	CADMIUM	0.400		0.407	mg/L	102	{
02	9231974	A228372	CADMIUM	0	0.050	0.0415	mg/L	83	4
SP102	0231973	A228372	CADMIUM	o	0.050	0.0398	mg/L	80	
C~: 01	D239279	NA	CADMIUM	0_0100		0.0116	mg/L	116	
€ .	0239277	INORGANIC	CADMIUH	5.00		4.48	mg/L	90	
BCA01	0239276	NA	CADMIUM			< 0.002	mg/L		\
LC\$	0231971	NA	CADMITUM	0.500		0.407	mg/L	81	
8 02	9231972	NA	CADMIUM	ļ		< 0.002	mg/L		İ
S PLE	A228370		See Certificate of An	ntyeis			1		}
CCV	0239275	INORGANIC	CADMIUM	5.00		4.80	mg/L	96	1
8)1	9239274	NA AK	CADMIUM			< 0.002	mg/L	1 ,	
a 11	9239270	NA	CADMIUM	0.0100		0.0122	mg/L	122	l

CHRONIUM ICP SW846-6010 matyet: M. JAD REVIEWER: S. ENDERSEN Prep: FAA OR ICP ACID			Analysis Bate: (Analysis Date: 01-MAY-91 Instrument: ICP Review Date: 02-MAY-91 File ID: DIGESTION OF AQUEOUS SAMPLES SW846-3005			Test:: N110;3.0 Run:: R119838			
Qt ype	identifier	Source	Parameter	True/Sampl	Spike Val	Deerved	Units	% Rec	RPD	
K H	q2 3925 4	WP1083	CHRONIUM	0.400		0.421	mg/L	105		
cvot	0239259	WP1083	CHROMEUM	0.400		0.400	mg/L	100		
21 10	0231974	A228372	CHROMTUM	0	0.200	0.193	mg/L	97	3	
SF 12	0231973	AZ28372	CHRONTUM	0	0.200	0.188	mg/L	94		
:DL01	9239279	NA	CHROMIUM	0.0200		0.0260	mg/L	130		

Page 1

QC Typel I	dentifier Source	Parameter	True/Sampi Spike Val	Observed	Units	% Rec	RPD
ηCΛ	3239277 INORGANIC	CHRONIUM	\$.00	4.54	mg/L	91	
.A01	9239276 NA	CHROMIUM		< 0.004	mg/L		
TTS	9231971 INA	CHROM1UM	2.00	1.85	mg/L	92	
8LA02	0231972 NA	CHROMIUN		< 0.004	mg/L		
MPLE	A228370	See Centificate of Analysis		1	i		
ν	0239275 INORGANIC	CHROMIUM	5.00	4.83	.mg/t	97	1
BLA01	9239274 NA	CHROMIUM		< 0.004	mg/L		
01	2239270 NA	CHROMIUM	0.0200	0.0210	mg/L	105	

NICKEL ICP SW846-6010

Analyst : M. JAO

Analysis Date: 01-MAY-91 Instrument: ICP

Test: M122.3.0

Reviewer: S. ENDERSEN

Review Date: 02-MAY-91 File ID: Run: R119838

Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES	SW846-3005	
--	------------	--

OC Type	Identifier	Source	Parameter	True/Sampl	Spike Val	Observed	Units	% Rec	RPD
v01	9239254	WP1083	NICKEL	0.400		0.419	mg/L	105	1
V01	9239259	WP1083	NICKEL	0.400		0.408	mg/L	102	
0PS02	0231974	A228372	NICKEL	0	0.500	0.468	mg/L	94	3
201	2231973	A228372	NICKEL	0	3.500	0.457	mg/L	91	!
L01	9239279	NA	NICKEL	0.0200	:	0.0229	mg/L	115	1 1
CCV	9239277	INORGANIC	NICKEL	5.00	· İ	4.50	mg/L	90	
BLAGT	0239276	NA	NICKEL	1		< 0.004	mg/L		
5	q2 31971	NA	NICKEL	5.00		4.62	mg/L	92	
A02	9231972	NA	NICKEL		Į	< 0.004	mg/L		į l
SAMPLE	A228370		See Certificate of Analysis						
1	9239275	INORGANIÇ	NICKEL	5.00		4,79	mg/L	96	
101	9239274	HA	NICKEL	1	<u> </u>	< 0.004	mg/L		
CDL01	0239270	NA	NICKEL	0.0200	l	0.0199	mg/L	99	<u>L</u>

5717	/FR	TCP	SWR	l6.	5010

Analyst : M. JAO

Analymis Date: 01-MAY-91 Instrument: ICP

Test: M130.3.0

Reviewer: S. ENDERSEN

Review Date: 02-MAY-91 File ID: Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES SW846-3005 Run: R119838

Type	Identifier	Source	Parameter	True/Sampl	Spike Val	Observed	Units	% Rec	RPD
10001	0239253	WP1083	SILVER	2.00		2.05	ng/L	102	
1 102	2231974	A228372	SILVER	0	0.050	0.0492	mg/L	98	2
:02	q2 31973	A228372	SILVER	0	0.050	0.0479	mg/L	96	
CDL01	0239252	NA.	SILVER	0.0200	1	0.0200	mg/L	100	
כרע	Q23 927 7	INORGANIC	SILVER	1.00		0.912	mg/L	91	
£ 01	0239276	NA .	SILVER			< 0.004	mg/L		†
Lus	Q231971	NA	BILVER	0.500		0.468	mg/L	94	i
BLAOZ	9231972	NĀ	SILVER	• • •	į	< 0.004	mg/L		1
S IPLE	A228370		See Certificate of Analysis		1		1	İ	1
'(_'	9239275	INORGANIC.	SILVER	1.00	Ì	0.950	mg/L	95	1
BLA01	0239 274	NA	SILVER			< 0.004	mg/L		
C 01	q239271	NA:	SILVER	0.0200	}	0.0208	mg/L	104	<u> </u>

IRON ICP SW846-6010

eviewer: S. ENDERSEN

Analyst : M. JAO

Analysis Date: 01-MAY-91 Instrument: ICP

Review Date: 02-MAY-91 File ID:

Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES SW846-3005

Test: M115.3.0 Run: R119838

; a	Туре	identifier	Source	Parameter	True/Sampl	Spike Val	Observed	Units	% Rec	RPD
ı	01	Q239254	WP 1083	IRON	0.400		0.425	mg/L	106	
ı	31	0239259	UP1083	IRON	0,400		0.413	Mg/L	103	ļ
DI	202	9231974	A228372	IRON	0	1.00	0.968	mg/L	97	3
\$'	-02	9231973	A228372	FROM	8.	1.00	0.942	.mg/L	94	ĺ
C	01	0239279	NA	IRON	0.0400	•	0.0407	mg/L	102	ĺ
Ç	V.	9239277	INORGANEC.	IRON	5.00		4,59	mg/L	92	

Page 2

APPENDIX B.6 EMS HERITAGE LABORATORIES, INC. PERFORMANCE AND SYSTEMS AUDITS

Section 14.0
Revision 0
Nov. May 1, 1991
Page 1 of Eg

14.0 Performance and Systems Audits

14.1 Performance Audits

A <u>performance audit</u> is an independent check to evaluate the data produced by a laboratory's analytical system, and may be categorized as a <u>quantitative appraisal of quality</u>. There are several ways that this is done:

- 1. Worksheet review
- 2. Oral worksheet review
- 3. On-site analyst work review
- 4. Independent or check sample review
- 5. Intra and interlaboratory check sample, or proficiency test (performance evaluation) sample analysis review

14.1.1 Internal Performance Audits

Internal performance audit worksheet reviews are conducted by the QA Officer and/or the QA Unit. These reviews must be conducted at a frequency necessary to assure the accuracy of either the total measurement system or its component parts. Worksheet reviews by the QAO and/or QAU will be ongoing but will occur at a minimum frequency of semi-annually for selected tests. This will be in addition to (and a more in-depth review of all data records) the 2-5 percent frequency of data entry validation performed by the QAU.

Internal performance audit worksheet reviews will consist of evaluations of all data and related supporting documentation to assure that all required QC checks are being made and evaluation criteria followed. Reports relating to internal performance audit worksheet reviews are confidential and will be released only upon approval from the President of EMS Heritage.

14.1.2 Performance Audit-Proficiency Test Samples

Performance Audits at EMS Heritage also consist of analysis of independent or commercial check samples and participation in performance evaluation sample programs.

Section 14.0 Revision 0 Nov. May 1, 1991 Page 2 of 6

1 REference Materials And

Refer to table 14.0, Performance Evaluation Sample Programs Participation for the sources of reference materials used and the on-going performance audit participation.

All information generated from Performance Evaluation (P.E.) sample programs will be made available during systems audits or upon request. Blind samples and split samples may be submitted as deemed necessary by the QAO or the QAU.

14.2 Systems Audits

A <u>systems audit</u> is an on-site inspection and review of a laboratory's quality control system and may be categorized as a <u>qualitative appraisal of quality</u>. It will cover any or all of the operational quality control elements of the quality assurance program. Systems audits include but are not limited to the following:

- 1. Sample handling: receiving, custody, log-in, storage
- 2. Sample analysis: written SOPs and analytical methods, protocols
- 3. Records control
- 4. Documentation: bound notebooks where required; records of all sample handling and analytical procedures
- 5. Preventative maintenance: adequate records, procedures.
- 6. Proficiency testing
- 7. Personnel practices
- 8. Training
- 9. Workload
- 10. Instrumentation and facilities

14.2.1 External Systems Audits

Systems audits are performed frequently at EMS Heritage by State and Federal agencies as part of the participation in sample analyses for

Section 14.0 Revision 0 Nov. May 1, 1991 Page 1 of 1

TABLE 14. REFERENCE MATERIALS AND PERFORMANCE EVALUATION PARTICIPATION

Source	Туре	Frequency	Parameters	Division
APG	Unknowns	Monthly	All	All
EPA-WP	Unknowns	Semi-Annually	All	All
EPA-WS	Unknowns	Semi-Annually	All	All
North Carolina- Wastewater	Unknowns	Semi-Annually	Metals, General Chemistries	Charlotte
State of New York	Unknown	Quarterly	Ali	Indjana polis
State of New Jersey	Unknown	Semi-Annually	All	Indjanapolis
EPA Reference	Known	On-Going	Ali	All
APG	Known	On-Going	All	All
NIST	Known	On-Going		All
EPA QB- Inorganics	Unknown	Quarterly	Metals	Indianapolis
EPA QB- Organics	Unknown	Quarterly	Organics	Indy anapolis

Chemical waste Mynt.

Unknown

Quarterly

MAIL

Indianapolis, Chicago

governmental organizations. In addition, many of our commercial clients perform routine audits of EMS Heritage. Evaluation by other appropriate outside experts is to be performed in the event that regulatory personnel are not available. At a minimum, systems audits will occur annually. EMS Heritage Laboratories will welcome any external audit from an organization which currently or proposes to submit samples for analysis.

14.2.2 Internal Systems Audits

In order to verify that each laboratory division is performing according to the standards established by EMS Heritage Laboratories, Inc., an internal audit system has been established. Internal systems audits will be conducted no less frequently than annually. The auditor will be appointed by and directly responsible to the President of the corporation.

The President may appoint for any given audit the following staff:

- 1. Quality Assurance Officer
- 2. Quality Assurance Unit
- 3. Outside (Contracted) Auditor

EMS Heritage also encourages the individual groups at each division to perform a self-audit at periodic intervals to assess their overall operation.

These audits will be scheduled by the auditor and each laboratory director. All reports relating to internal systems audits will be treated as an internal document only; release to any outside entity will be only upon approval of the President of EMS Heritage.

To facilitate the procedure and make it consistent from division to division, a set of audit procedures and protocols has been developed and will be used consistently throughout the corporation. These forms will be used as a guideline and a tool for documentation of problems and to assure that all important areas are covered. The forms are not an end in themselves and will be subject to change/improvement by the QAO at his discretion in order to facilitate and improve the auditing process. Refer to the attached Internal Systems Audit Forms.

Section 14.0 Revision 0 Nov. May 1, 1991 Page 4 of &

14.2.3 Internal Systems Audits - Procedures and Protocols

Not all areas (service groups) will necessarily be audited during each visit. During each audit, at least one analytical run will be randomly selected from the service group(s) chosen. This run will be thoroughly checked using the audit checklist forms and any other pertinent audit questions.

Upon completion of the audit, the auditor will conduct an exit interview with the QA Unit and the Laboratory Director and any other personnel deemed appropriate. A copy of the completed audit forms as well as any supporting narrative will be given to the QA Unit and the Laboratory Director at that time or immediately upon completion. A copy of all of these materials will also be sent to the President, Vice President of Operations, Quality Assurance Officer and all QA Units of all EMS Divisions. This sharing of audit information with all divisions is to provide not only a report of findings but will also serve to point out potential deficiencies which may exist at the respective divisions not audited. The auditor will retain a copy of the materials to use in the next audit. The QAO will confer with the President to prioritize items for the next audit.

Generally the audit will consist of the auditor reviewing with the group leader and the analyst all the steps involved in generating a run of reportable data. It will also include an overview of the entire lab, address issues relevant to the operation as a whole (i.e. cooler logs, log-in procedures, over logs, etc.) Any deficiencies will be noted on the forms. Deficiencies will be discussed at the time they are noted and the auditor will explain what will be required to correct this deficiency.

Form 1 will always be filled out. It addresses general laboratory practices. Form 2 will be filled but during each audit for each service group audited. Forms 3-5 will be used on a service group specific basis only.

INTERNAL SYSTEMS AUDIT FORMS

Appendix G Revision 0 Nov. 1, 1991 Page 1 of 12

FORM	M 1	
DAT	E	
LOCA	ATION	
AUD	ITOR	
	GENERAL INFORMATION	
1)	Does the laboratory have copies of SOPs/methods manuals and QA Plans available to all personnel?	YES NO COMMENTS
2)	Are training records maintained and up to date? Is training by group leader or above?	
3)	Is a Sample Custodian designated?	
4)	Are written Standard Operating Procedures (SOPs) developed for receipt and storage of samples?	
5)	Are samples stored in such a way that their preservation is maintained?	
6)	Are refrigerator/freezer logs maintained, checked daily and up to date?	
7)	Are excursions in cooler temperature noted and appropriate actions taken as required?	
8)	Are volatiles stored separately from semi-vols?	
9)	Does someone responsible review and initial the sample log and log sheets daily?	
10)	Are contamination free areas provided for trace level analytical work?	
11)	Does the facility appear clean and safe?	
12)	Are toxics handled in fume hoods? Are fume hoods checked & documented quarterly?	
13)	Are the toxic chemical handling areas either a stainless steel bench or an impervious material covered with absorbent material?	
91GB114	18.153	

Appendix G Revision 0 Nov. 1, 1991 Page 2 of 12

PAGE 2 OF FORM 1

		YES	NO	COMMENTS
14)	Is there documented "trace-free" water available for preparing standards and blanks?			
15)	Is the conductivity of "trace-free" water routinely checked and recorded?			
16)	Is (Are) the analytical balance(s) correctly located (free from drafts and rapid temperature changes) and checked semi-annually by a certified technician and documented?			
17)	Are the balances checked with class S weights and documented at least weekly?			
18)	Are solvent storage cabinets properly vented in order to prevent possible laboratory contamination?			
19)	Are reagent grade (or higher purity) chemicals used to prepare standards and reagents?			
20)	Are reagents dated upon receipt and upon opening? Is First-in, First-out method used?			
21)	Are reagents standardized before use?			
22)	Generally, are reference materials properly labeled with concentrations, preparation date, solvent, preservative expiration date and name of person preparing?			
23)	Are spike/calibration stock standards preparation and tracking logbook(s) maintained?			
24)	Are the primary standards approved by the EMS QAP?			
25)	Are bench sheets filed so that they are readily accessible?			
26)	Are standards and samples (& extracts) stored separately?			
27)	Are samples checked for proper preservation upon arrival? Is this noted within LIMS?			
28)	Are logs maintained for all ovens and incubators?			
29)	Is an NBS thermometer available?			

Appendix G Revision 0 Nov. 1, 1991 Page 3 of 12

		YES NO COMMENTS
30)	Are copies of EPA - PEs and others such as APG on file?	
31)	Is the distilled/deionized water system functioning properly? Is it checked daily and noted in a logbook?	
32)	Are samples requiring chain of custody documentation properly checked before signing chain of custody papers?	
33)	What is done with lab waste?	
24		
34)	Are chemical waste disposal policies/procedures adequate?	
35)	Are blind samples introduced into the lab by the QAO?	
36)	Are corrections on bench sheets, lab books, etc. made in such a way that initial entries are legible?	
37)	Are such corrections initialed and dated?	

PAGE 3 OF FORM 1

Appendix G Revision 0 Nov. 1, 1991 Page 4 of 12

FOR	M 2	
DAT	E	
SER	VICE GROUP	AUDITOR
LOC	ATION	ANALYST
	TEST	T YES NO COMMENTS
11	Ass mathods magnets & OAD's smileble to the	
1)	Are methods manuals & QAP's available to the	analysis:
2)	Is the SOP for glassware washing posted at stations?	t the cleaning
3)	Are the types and numbers of required Blanks b	peing checked?
	Bia 01? Bia 02? Cal 00?	
4)	Are blank data logged to the QA sheet and t LIMS?	transferred to
4a)	Does blank data appear to be "in control"?	
4b)	Do original instrument outputs agree with what	was reported?
5)	Are calibration curves maintained for all analyte	rs?
6)	Are calibrations verified (1 point) or performed (5 to the analysis of samples CAL01 + CAL00? Comment:	<u> </u>
7)	Is an EPA, NIST or other approved external references and verify concentrations of CAL standar result recorded on the QA sheet?	
7 a)	Are the results recorded in LIMS?	
8)	Do the analysts record bench data in a neat manner?	and accurate

Appendix G Revision 0 Nov. 1, 1991 Page 5 of 12

YES	NO	COMMENTS
	YES	YES NO

Appendix G Revision 0 Nov. 1, 1991 Page 6 of 12

PAGE 3 OF FORM 2

		AFZ	NO	COMMENIS
19)	Are standard recoveries correctly calculated? Are spike recoveries correctly calculated? Are duplicate recoveries correctly calculated? Are they all documented in LIMS?			
20)	Does a manual recalculation agree with concentrations otherwise calculated and/or reported? (is verify electronic integrator performing and correct peaks were chosen for GC, GC/MS)			
21)	For GC/MS, are unknowns (non-target compounds) correctly searched in the NBS library and documented as present with an estimated concentration?			
22)	For ICP, are interelement interference check solutions properly analyzed and documented prior to each run?			
23)	Have detection limits been empirically determined according to 40CFR136 for the analytes determined?			···
24)	Are standards correctly Labelled? Logged & documented?			
25)	Are instrument operating manuals readily available?			
26)	Are data acceptance criteria developed and used for Blanks? Duplicates? Verification standards? Spikes?			
27)	Are instrument maintenance logs in place and maintained?		T	
28)	Is the mercury analyzer operational and well maintained (i.e. properly vented)?			
29)	Is preventive maintenance applied and documented?			
30)	Are data calculations spot-checked by a second person? Does this person initial these checked calculations?			

Appendix G Revision 0 Nov. 1, 1991 Page 7 of 12

PAGE 4 OF FORM 2

YES NO COMMENTS

31)	Do supervisory personnel review beach sheets and initial them?	
32)	Do records indicate that corrective action was taken as necessary?	
33)	Has a cooperative attitude been displayed?	
34)	Has corrective action indicated during previous visits been implemented?	
35)	Are extractions/digestion performed within holding times?	
36)	Are analyses performed within holding times?	
37)	Are sample preparation methods correct?	
38)	Is a background corrector properly used? (AA, GFAA, ICP)	
39)	Is an approved methodology used for the runs being examined?	
40)	Have acceptance criteria for start-up QC been met?	
41)	Do instrument outputs (strip charts, print-outs, bench sheets, etc.) agree with what is in the final report?	
42)	Are labs data validation procedures adequate?	
43)	Based upon blank, standard, spike and duplicate data, are goals of data accuracy and precision being met?	
44)	Is the method of standard additions being used correctly for HGA analyses?	
45)	Is analytical sensitivity adequate?	
46)	Are instrument guidelines and/or acceptance criteria used at the bench and at data review which determine the acceptability and reportability of data?	
47)	Are Blanks carried through entire process?	
48)	Are desiccants changed and documented?	

Appendix G Revision 0 Nov. 1, 1991 Page 8 of 12

m	•			ΛE	EVA	TOB	•	•
r	А	G-L	3	OF	ΓU	KI	л	Z

49) Are TOC, TOX and BTU instrument logs current?

50) Are normalities of titrants routinely checked?

51) Are sufficient cyanide distillation apparatus available to routinely analyze all samples within the required holding period?

Appendix G Revision 0 Nov. 1, 1991 Page 9 of 12

	FORM 3	
	LAB	
	DATE	
	AUDITOR	
	METALS GROUPS	YES NO COMMENTS
1)	Are samples for mercury analysis collected in glass and preserved with HNO ₃ and Permanganate?	
2)	Are mercury determinations performed within 28 days of sample collection?	
3)	Are proper sample digestion techniques used and documented?	
4)	Is background correction used a) For all HGA work? b) As required for flame AA work?	
5)	Are correct matrix modifiers used for HGA?	
6)	Is an ionization suppressor used as needed for flame AA?	
7)	For ICP are interelement corrections checked?	
8)	Do prep sets of 10 include proper QA and acceptance criteria?	
9)	Are wavelengths used recorded on bench sheets?	
10)	Are flame/furnace/ICP programs adequate? a) Integration times adequate? b) Furnace temperature programs correct? c) Sample sliquot or deposition times adequate?	
11)	Are sensitivity guidelines available at instruments?	

Appendix G Revision 0 Nov. 1, 1991 Page 10 of 12

FOR	M 4	
LAB		
DAT	E	
AUD	ITOR	
	GAS CHROMATOGRAPH	YES NO COMMENTS
1)	Are samples collected in amber glass bottle with teflon-line lids?	od
2)	Are samples kept at 4°C	
3)	Are samples extracted within 7 days?	
3 a)	Analyzed within 40 days?	
4)	Are standards properly documented?	
5)	Are standards prepared fresh regularly?	
6)	Is there a well maintained log, documenting stability of a detectors?	
7)	Are Arochior 1221 and 1232 standards run at the proper frequency and the data maintained for on-site inspection?	er

Appendix G Revision 0 Nov. 1, 1991 Page 11 of 12

FORM 5				
LAB				
DATE				
AUDITOR				
	GC/MS	YES	NO	COMMENTS
1)	Are samples extracted and/or analyzed within all required holding times?			
2)	Are control limits established and used for CCC and SPCC samples?			
3)	Is QA data properly transferred to LIMS?			
4)	Are Blanks, Spikes and Standards analyzed at the appropriate frequency?			
5)	Is Spike/spike duplicate data being generated?			
6)	Are surrogates properly analyzed with all samples?			
7)	Are surrogate results tabulated?			
8)	Are both 5 and 25 ml purge vessels available and properly used?			J
9)	Are correct internal standards being used?			
10)	Is data reviewed prior to issuing final report?			
11)	Has QAO reviewed 2-5% of the runs?			
12)	Are reviewed bench sheets initialed?		1	
13)	Are all tuning criteria met?		1	_
14)	Is the trap for the purge and trap filled with the proper adsorbent?			
at/201149 IS2				

Appendix G Revision 0 Nov. 1, 1991 Page 12 of 12

PAGE 2 OF FORM 5

- 15) Is raw data being archived and documented properly (i.e magnetic tape)?
- 16) Is a split/splitless capillary injector in place?

YES	NO	COMMENTS	
<u> </u>	1	r	
			ļ
}			
ļ			

APPENDIX C LANCASTER LABORATORIES, INC. QUALITY ASSURANCE PLAN

Section No. 1 Revision No. Date: 11/14/91 Page 1 of 1

Laboratory Quality Assurance Plan

This document provides the laboratory portion of the response to EPA's "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" QAMS-005/80, Sections 5.1 - 5.16 as revised December 29, 1980.

As much as possible, the procedures in this document have been standardized to make them applicable to all types of environmental monitoring and measurement projects. However, under certain site specific conditions, all of the procedures discussed in this document may not be appropriate. In such cases it will be necessary to adapt the procedures to the specific conditions of the investigation.

Director of Quality Assurance M& Mission (180)

Section No. 2 Revision No. 1 Date: 3/16/92 Page 1 of 1

Sect	ion	Pages	Revision	Date
1.	Title Page	1		
2.	Table of Contents	1	1	3/16/92
3.	Project Description	1	•	
4.	Project Organization and Responsibility	4		
5.	QA Objectives for Measurement Data, in terms of precision, accuracy, completeness representativeness and comparability	2		
6.	Sampling Procedures	2	1	3/16/92
7.	Sample Custody	9		
8.	Calibration Procedures and Frequency	3		
9.	Analytical Procedures	20	1	3/16/92
10.	Data Reduction, Validation and Reporting	5		
11.	Internal Quality Control Checks	4		
12.	Performance and Systems Audits	15		
13.	Preventive Maintenance	2		
14.	Specific Routine Procedures Used to Access Data Precision, Accuracy and Completeness	3		
15.	Corrective Action	3		
16.	Quality Assurance Reports to Management	1		
Appe	endix A - Reporting Forms	10	1	3/16/92

Section No. 3
Revision No.
Date: 11/14/91
Page 1 of 1

3. Project Description

Tests will be performed according to the analytical methodology set forth in Section 9. These OSHA and NIOSH references provide specific analytical procedures to be used and define the specific application of these procedures. The soil vapor samples will be analyzed for selected Volatile Organic Compounds and Phenol. Proven instruments and techniques will be used to identify and measure the concentrations of all analytes. The laboratory will employ state-of-the-art procedures to perform all organic analyses, including all necessary preparation for analysis. The client is responsible for providing specifics on the project site.

Section No. 4
Revision No.
Date: 11/14/91
Page 1 of 4

4. Project Organization

The objectives of the laboratory Quality Assurance Program are to establish procedures which will ensure that data generated in the laboratory are within acceptable limits of accuracy and precision, to ensure that quality control measures are being carried out, and to ensure accountability of the data through sample and data management procedures. To this end, a Quality Assurance Department has been established. The Director of Quality Assurance reports directly to the President of the Laboratory and has no direct responsibilities for data production, thus avoiding any conflict of interest.

The attached organizational charts show the key personnel in both Corporate Services and the Environmental Sciences Division. Resumes of key individuals may be found in the enclosed Qualification Manual.

The Sample Administration Group will be responsible for receiving samples, signing the external chain-of-custody, checking sample condition, assigning unique laboratory sample identification numbers, assigning storage locations, checking and adjusting preservation, and homogenizing the sample as needed.

Group Leaders listed in each technical area are responsible for performing laboratory analyses, quality control as specified in the methods, instrument calibration, and technical data review. Data is reported using a computerized sample management system, which tracks sample progress through the laboratory and generates client reports when all analyses are complete. Quality control data is entered onto the same system for purposes of charting and monitoring data quality.

Section No. 4
Revision No.
Date: 11/14/91
Page 2 of 4

The Quality Assurance Department is responsible for reviewing quality control data, conducting audits in the laboratory and reporting findings to management, maintaining current copies of all analytical methods, maintaining copies of computer code used to calculate and report results, submitting blind samples to the laboratory and ensuring that appropriate corrective action is taken when quality problems are observed.

Data package deliverables are available upon request. The Quality Assurance Department reviews the contents of the deliverables for completeness and to be sure that all quality control checks were performed and met specifications. This step includes review of holding times, calibrations, instrument tuning, blank results, duplicate results, matrix spike results, and surrogate results. Every attempt to meet specifications will be made and any item outside of the specifications will be noted in the narrative. The laboratory will not validate data with regard to useability since this generally requires specific knowledge about the site.

Revision No. Date: 11/14/91 Lancaster Laboratories, inc. Page 3 of 4 Corporate Services Earl Hess, Ph.D. President Chief Executive Officer **Council of Vice Presidents** Fred Albright, Ph.D. Wilson Hershey, Ph.D. Kenneth Hess, B.S. Carol Miller, M.B.A. Louise Hess, B.S. Director, Quality Assurance Anne Osborn, M.A. Assistant to the President Fred Albright, Ph.D. Wilson Hershey, Ph.D. Carol Miller, M.B.A. Kenneth Hess, B.S. Senior Vice President/Director

Vice President

Chief Financial Officer

Vice President/Director

Environmental Sciences

Food and Pharmaceutical

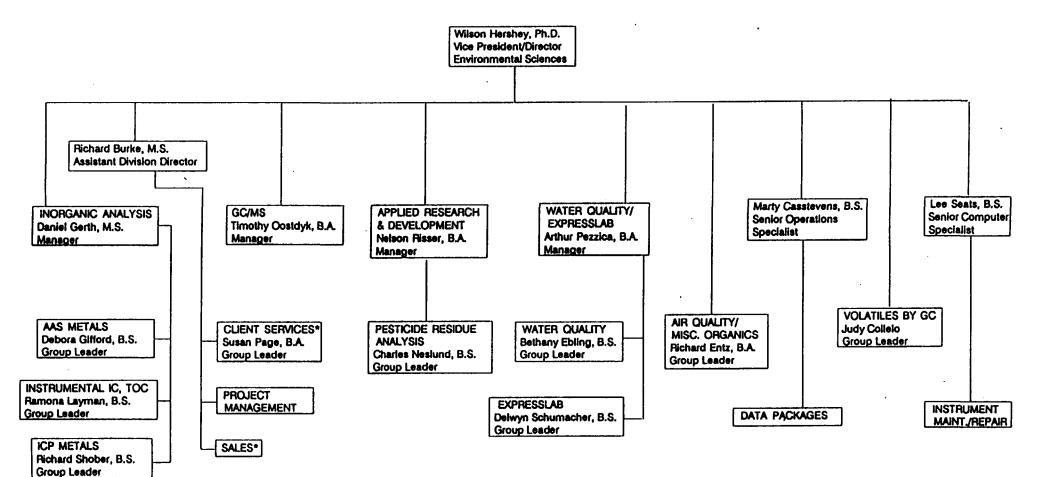
Seccion No. 4

Vice President

Director, Administration

Section No. 4
Revision No.
Date: 11/14/91
Page 4 of 4

Lancaster Laboratories, Inc. Environmental Sciences



^{*}These groups cover client services and sales for all technical operations.

Section No. 5
Revision No.
Date: 11/14/91
Page 1 of 2

5. QA Objectives For Measurement Data

Quality Assurance is the overall program for assuring reliability of monitoring and measurement data. Quality control is the routine application of procedures for obtaining set standards of performance in the monitoring and measurement process. Data quality requirements are based on the intended use of the data, the measurement process, and the availability of resources. The quality of all data generated and processed during this investigation will be assessed for Precision, Accuracy, Representativeness, Comparability, and Completeness.

Precision - Precision is determined by measuring the agreement among individual measurements of the same property, under similar conditions. The laboratory objective is to equal or exceed the precision demonstrated for the applied analytical method on comparable samples. The degree of agreement is expressed as the relative percent difference (RPD%). Evaluation of the RPD% is based on statistical evaluation of past lab data for organic and inorganic analyses. External evaluation of precision is accomplished by analysis of Standard Reference Material and interlaboratory performance data.

Accuracy - Accuracy is a measure of the closeness of an individual measurement to the true or expected value. Analyzing a reference material of known concentration or reanalyzing a sample which has been spiked with a known concentration/amount is a way to determine accuracy. Accuracy is expressed as a percent recovery (%R). Evaluation of the %R is based on statistical evaluation of past lab data or guidelines within the methods for organic and inorganic analyses.

Section No. 5
Revision No.
Date: 11/14/91
Page 2 of 2

Representativeness - Representativeness expresses the degree to which data accurately represents the media and conditions being measured. The representativeness of the data from the sampling site will depend on the sampling procedure. Sample collection is the responsibility of the client. Samples will be homogenized, if required, as part of the laboratory sample preparation. By comparing the quality control data for the samples against other data for similar samples analyzed at the same time, representativeness can be determined for this objective.

Comparability - Comparability conveys the confidence with which one set of data can be compared to another. The analytical results can be compared to other laboratories by using traceable standards and standard methodology and consistent reporting units. The Laboratory Quality Assurance Program documents internal performance, and the interlaboratory studies document performance compared to other laboratories.

Completeness - Completeness is a measure of the quantity of valid data acquired from a measurement process compared to the amount that was expected to be acquired under the measurement conditions. The completeness of an analysis can be documented by including in the data deliverables sufficient information to allow the data user to assess the quality of the results. Additional information will be stored in the laboratories archives, both hard copy and magnetic tape. Quality Assurance Standard Operating Procedures (SOP's) are in place to provide traceabilty of all reported results.

Section No. 6
Revision No. 1
Date: 3/16/92
Page 1 of 2

6. Sampling Procedures

In order for meaningful analytical data to be produced, the samples analyzed must be representative of the system from which they are drawn. It is the responsibility of the client to ensure that the samples are collected according to accepted or standard sampling methods.

Specific requirements for the collection, preservation, and handling of Soil Vapor Samples follows:

Section No. 6
Revision No. 1
Date: 3/16/92
Page 2 of 2

Preservation and Handling of Soil Vapor Samples

Air samples for the volatile organics will be collected on coconut charcoal sorbant tubes, 150 mg charcoal per tube (SKC part number 226-01). The tubes will be supplied by Lancaster Laboratories, Inc. as sealed by the manufacturer. In the field the ends will be cut off; the tubes assembled in series with a minimum of connecting tubing between the tubes; the sampling flow at 0.2 liters per minute; the volume of air drawn through the tubes (approximately 10 liters); the ends capped with the supplied plastic caps; and identified with the sampling location, time, temperature, and volume flow rate. The tubes will be sealed in individual plastic bags and placed a screw top glass jar with coconut charcoal and stored and shipped under refrigerated conditions, 4°C, and returned to the laboratory within two days.

In the laboratory, the samples will be kept at 4°C until the analysis is begun. The analysis must be performed within seven days of receipt by the laboratory.

Air samples for phenol will be collected on XAD-7 sorbant tubes, 100 mg front section + 50 mg back section (SKC part number 226-30-12-07). The tubes will be supplied by Lancaster Laboratories, Inc. as sealed by the manufacturer. In the field the ends will be cut off; the sampling flow set at 0.1 liters per minute; the volume of air drawn through the tubes (approximately 10 liters); the ends capped with the supplied plastic caps; and identified with the sampling location, time, temperature, and volume flow rate. The tubes will be sealed in individual plastic bags and placed in a screw top glass jar with coconut charcoal and stored and shipped under refrigerated conditions, 4°C, and returned to the laboratory within two days.

In the laboratory, the samples will be kept at 4°C until the analysis is begun. The analysis must be performed within fourteen days of receipt by the laboratory.

Section No. 7
Revision No.
Date: 11/14/91
Page 1 of 9

7. Sample Custody

٠.

A member of our Sample Administration Group will act as sample custodian for the project. To ensure accountability of our results, a unique identification number is assigned to each sample as soon as possible after receipt at the laboratory. When samples requiring preservation by either acid or base are received at the laboratory, the pH will be measured and documented. Samples requiring refrigeration will be stored in our walk-in cooler which is maintained at 4°C. The use of our computer system in tracking samples (by the LLI sample # assignment) will control custody of the sample from receipt until the time of its disposal. The security system on our laboratory building allows us to designate the entire facility as a secure area since all exterior doors are either locked or attended. Therefore, hand-to-hand chain of custody is not part of our routine procedure but, is available upon request. The procedures for sample log-in and chain-of-custody documentation are detailed in the QA Standard Operating Procedures included in Section No. 7 (QA102 and QA104).



<u>Initiated Date: 3/87</u> Revised Date: 5/16/90

QUALITY ASSURANCE OPERATIONS MANUAL STANDARD OPERATING PROCEDURE QA-102

Title: Sample Log-in

Purpose:

In order to provide accountability of our results and to prevent sample loss or mix-up, a unique identification number is assigned to each sample.

Scope:

This SOP will cover the procedure used to log-in samples received for analysis.

Procedures:

- All samples received by laboratory personnel shall be delivered to the Sample Administration Group immediately upon arrival at the laboratory.
- 2. All client correspondence relating to samples shall also be transferred to the Sample Administration Group. This includes purchase orders, quotes, letters and completed entry request forms.
- 3. Personnel of the Sample Administration Group shall log the samples into the computer as soon as practical after receipt. The computer will assign a unique identification number to each sample. Samples shall be logged in on the same day they are received with the following exceptions:
 - a. Samples received during a holiday or between 6 p.m. on Friday and 6 p.m. on Sunday. These samples shall be logged-in on the next normal work day.
 - b. Samples submitted by clients without any indication of the tests to be performed or with unclear or incomplete information. Every effort shall be made to contact the client on the same day as sample receipt.

If same day entry is not possible, any special storage requirements (e.g., refrigeration) should be observed.

Section No. 7 Page 3 of 9

SOP-QA-102

Initiated Date: 3/87 Revised Date: 5/16/90

Page 2 of 2

4. Upon assignment of a sample number, the computer will generate a label which shall be attached to the sample container. The information on the label will include the LLI sample number, the client name, the storage location, a list of analyses requested (by analytical method number), a bottle code indicating container and preservative type, and a unique bar code.

- 5. Addition of preservatives to unpreserved samples will be the responsiblity of the Sample Administration Group. Preservation should be performed immediately after log-in. A list of preservatives required for routine analyses may be found in the Fee Schedule.
- 6. All entries in preservation notebooks and on client paperwork shall be made in ink. The error correction procedure given in SOP-QA-109 shall be followed for any changes made in this documentation.
- 7. After samples are logged-in (or preserved, if required) they shall be stored in the computer-assigned location. If the computer-assigned location is inappropriate for the samples, the location code may be changed by manually overriding the computer.

QA102 SOP QA #1

Prepared by:	M Lauise Le.	<i>\(\omega_{</i>	Date:	5/21/90
Approved by:		$\overline{}$	Date:	4 Vane Ge
Read and underst	ood by:		Date:	



Initiated Date: 3/87 Revised Date: 9/28/90

QUALITY ASSURANCE OPERATIONS MANUAL STANDARD OPERATING PROCEDURE QA-104

Title: Chain-of-Custody Documentation

Purpose:

In order to demonstrate reliability of data which may be used as evidence in a legal case or required by a regulatory agency, an accurate written record tracing the possession of the sample from its receipt at the laboratory to the time of its disposal must be maintained.

Scope:

Procedures for initiating and maintaining chain-of-custody documentation are described in this document.

Definition:

A sample is in custody if it is in any one of the following states:

- 1. In actual physical possession.
- 2. In view after being in physical possession.
- In physical possession and locked up so that no one can tamper with it.
- 4. In a secured area, restricted to authorized personnel.

Procedures:

1. Chain-of-custody documentation shall be kept upon request of the client or for any samples which are known to be involved in a legal dispute. As with all analytical data, it is extremely important that documentation be filled out completely and accurately with every transfer. If changes to the form need to be made, the error correction procedure given in SOP-QA-109 shall be followed.

Section No. 7 Page 5 of 9

SOP-QA-104
Initiated Date: 3/87
Revised Date: 9/28/90

Page 2 of 4

2. If requested by the client, the chain-of-custody documentation will begin with the preparation of bottles. A form (see Attachment 1) will be initiated by the person packing the sample bottles for shipment to the client. If the delivery of bottles is via our Transportation Department, the driver shall sign the form when relinquishing the bottles. Drivers must also sign chain-of-custody forms when picking up samples which require such documentation.

- 3. When samples arrive at the laboratory, a member of the Sample Administration Group will receive them and sign the chain-of-custody form, if one is provided with samples. If the sample was picked up by our Transportation Department, the driver must sign to indicate relinquishing the sample.
- 4. Samples will be logged into the computer as described in QA-102. Sample Administration personnel shall indicate locked storage, enter a lab note to inform analysts of the need for chain-of-custody documentation, and enter the analysis number for "laboratory chain-of-custody".
- 5. Sample Administration personnel shall initiate a "Laboratory Chain-of-Custody" form (Attachment 2) for each type of container in the sample, and relinquish the samples to a sample custodian or designated key holder, who will store the sample in the assigned locked location. At this point, external chain-of-custody forms will be filed with the Accounts Receivable Department to be returned with the invoice, and the internal forms will accompany the samples.
- 6. Sample handling should be kept to a minimum. Analysts requiring use of a sample will requisition it through the computer requisition program. During the hours where sample support is manned by sample custodians, the custodian will receive the computerized requisition, remove the sample from storage and sign the "released by" column to indicate the sample has been relinquished. The analyst shall sign the "received by" column and note the reason for change of custody before taking the samples to their work area. It will be a shared responsibility of technicians and sample custodians to ensure that forms are signed with each transfer.

Section No. 7 Page 6 of 9

SOP-QA-104

Initiated Date: 3/87 Revised Date: 9/28/90

Page 3 of 4

All changes of custody must be documented on the form. The following changes of custody shall be handled as follows:

- a. Signatures involving transfers from one shift to another shall be the responsibility of the technician who originally acquired the sample from sample support. When samples are then returned to storage, the person returning the samples shall be responsible to sign the "released by" column, and to ensure that samples were properly received by the custodian with his/her signature in the "received by" column.
- b. Occasionally a sample will be needed for analysis by a technician in a department while it has been signed out to a technician in another department. It will be the responsibility of the first technician who received the sample to see that the second technician needing the sample signs for receipt and return of the sample to them.
- c. Weekend work hours do not always have a sample custodian available. During these times the Lancaster Labs security personnel function as key holders to the storage areas. Technicians requiring use of samples over these times must obtain signatures from security personnel, in place of regular sample custodians. It may be necessary to page the security staff on weekends to acquire their signatures and assistance.
- d. Some samples are released by sample support and stored temporarily in other areas of the laboratory e.g. GC/MS Volatiles. During this time they may be worked on by several people in that department. Each of these people must sign for change of custody. These samples when completed are then returned to sample support. It will be the responsibility of the department who held temporary storage to see that all necessary signatures are on the chain of custody form before returning samples and forms, at the same time, to sample support. It is also important to return these sample groups as soon as possible after verification of data, because the chains may be required for data packages.

Section No. 7 Page 7 of 9

SOP-QA-104

Initiated Date: 3/87 Revised Date: 9/28/90

Page 4 of 4

7. Analysts in possession of samples shall remove the aliquot required for analysis and return the sample to storage as described in #8 below with a minimum of delay. During the time of possession, samples must remain in the analyst's view or be locked-up. If additional containers of the sample are created (e.g., an extract container from preparation for organic analysis), an additional form marked with the container type shall be created to accompany the new container.

- 8. After analysis, samples shall be relinquished to a key holder or sample custodian who will return the samples to locked storage. The forms which remain with the samples shall be signed again to indicate storage, and the sample custodian shall review the forms to ensure that all transfers are completely documented. Sample custodians shall not return a sample to its storage location without signing an accompanying chain.
- 9. After completion of analysis, these forms are given to the Data Package Group for inclusion in extended reports.

QA104 SOP QA #1

Prepared by: M. Luise des	Date:	10/2/90
Approved by: Ellantly	Date:	2.Octgo
Read and understood by:	Date:	

Section No. 7 Page 8 of 9



Locked Storage Internal Chain of Custody

ORIGINAL SAMPLE

Client/Project	:t:											
Preservative:			Matrix:Analyses:									
Sample # Rang	Sample # Range:											
Storage Location:												
Sample Number(s)	Released by	Received by	Date	Time	Reason for Change of Custody							
•												
	!											
				 								
				 								
				 -								
				 								
		 			+							
		<u> </u>		-								
	<u> </u>	<u> </u>			<u> </u>							

This form has been designed to accompany the sample from the moment it is originally entered into the computer until the last test is verified.

2016 Rev. 12/19/89

Ancaster Laboratorie
INCORPORATE

2425 New Holland Pike Laborator, RA 17801 & 717/058-200

Chain of Custody

Lancaster Laboratories Sample Number								1	Sample Type:						
Client:	Client: P.O. No.:									·	HZ Hazardous SO Soil				
Work Order No.: Project Name:									PW Potable Water GW Ground Water						
Submit Report to: FSC:							sw	Surface Water							
Sampler:		Project Location:					Analysos					WW SL	Waste Water Sludge		
Field sample number / sample identification	Date	Time	COMP	GR AB	Number of Containers (Total)	Same	A TAY LON						Remar	ks:	
			-	_			-								
			-				-				 	·			
							-								
			_												
		· ·	_												
				_											
0															Page
Sample Relinquished by:	Date	Time			Samplo Received I	by:		-	Date	Tin	ne		Rea	son for Transfer	Page 9 of 9
										ļ					7
	-							-							
	1	L	L		· · · · · · · · · · · · · · · · · · ·										

Section No. 8
Revision No.
Date: 11/14/91
Page 1 of 3

8. Calibration Procedures

Procedures for initial calibration and continuing calibration verification are in place for all instruments within the laboratory. The calibrations generally involve checking instrument response to standards for each target compound to be analyzed. The source and accuracy of standards used for this purpose are integral to obtaining the best quality data. The standards are purchased from commercial supply houses either as neat compounds or as solutions with certified concentrations. The accuracy of these purchased standards is checked by comparing to solutions obtained from USEPA, when available. solutions and all neat materials require subsequent dilution to an appropriate working range. All dilutions performed are documented and the resulting solution is checked by obtaining the instrument response of the new solution and comparing with the response to the solution currently in use. Any discrepancies between the responses are investigated and resolved before the new solution is used. Each standard is assigned a code which allows traceability to the original components. The standard container is marked with the code, date prepared and the initials of the preparer. Shelf-life for standards are included in the calibration procedures and new standards are prepared before the expiration date.

Section No. 8
Revision No.
Date: 11/14/91
Page 2 of 3

Each instrument is calibrated with a given frequency using one or more concentrations of the standard solution. As analysis proceeds, the calibration is checked for any change in instrument response. If the calibration check verifies the initial response, the analysis proceeds. If the calibration check indicates that a significant change in instrument response has occurred, then a new calibration is initiated. If necessary, maintenance may be performed prior to the recalibration.

Calibration records are usually kept in the form of raw data with the other instrument print-outs. In cases where no data system is used, calibration data is manually recorded in notebooks. Any maintenance or repair is also recorded in a notebook. The information recorded either in the notebooks or on the instrument print-out includes the date, employee name and/or identification number, and concentration or code number of standard.

The frequency of calibration and calibration verification, number of concentrations used, and acceptance criteria for each of the instruments to be used are listed on Table 8-1.

Section No. 8 Revision No. Date: 11/14/91 Page 3 of 3

Table 8-1

Initial Calibration

Continuing Calibration Verification

Instrument	Frequency	# of Standard Concentrations	Acceptance Criteria	Freguency	# of Standard Concentrations	Acceptance Criteria
Gas Chromatograph (Volatiles)	Each Batch	5	RSD ≤ 25%	Every 10 Samples	1	X D ≤ 15X
HPLC (Phenol)	Each Run	5	Correlation Coefficient ≥ 0.995	Every 4 Samples	1	Calib. results will be averaged. Correlation coefficient for linear least squares fit >0.995

Section No. 9 Revision No. 1 March 16, 1992 Page 1 of 20

9. Analytical Procedures

The analytical procedures to be used for Volatile Organic Compounds listed in Table 9-1 are those described in NIOSH methods 1500 and 1003 (modified). Phenol will be determined according to OSHA method 32. Copies of these method are included in Section 9.

<u>Volatiles</u> - This method determines the concentration of volatile organic compounds in air. This method involves collection of the vapor sample onto charcoal tubes, desorption with carbon disulfide, and subsequent analysis by gas chromatography.

<u>Phenol</u> - This method is applicable to the analysis of phenol in air. The method involves collection of the vapor sample onto a sorbant tube, desorption with methanol, and subsequent analysis by high performance liquid chromatography (HPLC).

Table 9-1
Estimated detection limit for soil vapor study *

Analyte	Estimated LOQ**	Required		
<u>LOQ</u> ***	ppm (v/v)	ppm (v/v)		
Acetone	0.35	254		
Chlorobenzene	0.29	100 #		
Chloroform	0.46	496		
1,1-Dichloroethane	0.32	3.4		
1,1-Dichloroethylene	0.30	515		
Ethyl Benzene	0.13	42		
Methyl Ethyl Ketone	0.30	139		
Methyl Isobutyl Ketone	0.22	233		
Methylene Chloride	0.63	22.4		
Tetrachloroethylene	0.61	16.8		
Toluene	0.21	36556		
1,1,1-Trichloroethane	0.40	2819		
1,1,2-Trichloroethane	0.40	1.1		
Trichloroethylene	0.45	71.5		
Xylenes (all isomers)	0.20	4794		
Phenol	0.20	1.4		

^{*} Assumes a 10 liter sample volume and two 150mg charcoal tubes or one 100 mg/50 mg XAD-7 tube used for sampling.

^{**} Based on 10 times the MDL.

^{***} Based on the concentrations of soil vapor at equilibrium with acceptable soil concentrations.

[#] No specific concentration is listed for chlorobenzene.

Section No. 9 Revision No. 1 March 16, 1992 Page 3 of 20

Selected Organic Compounds on Charcoal Tube for ECC Site - Soil Vapor Analysis

References:

NIOSH Methods 1003, 1005, 1015, 1022, 1300, 1500 (Third Edition) and P&CAM 127 (Second Edition).

Principle:

Organic vapors in soil gas are collected on charcoal tubes by passing the gas through the charcoal tubes at a controlled rate for a specified period of time. The volatile organic compounds are removed from tube by desorbing the tube with carbon disulfide and analyzing the solvent by gas chromatography. Knowing the exact volume of soil gas passed through the tube and the mass of the organic compound sorbed on the tube, the concentration of the compound in the soil gas can be calculated.

Scope:

This method is applicable to the analysis of volatile organic compounds sorbed on charcoal tubes. Two charcoal tubes, each containing 150 mg of charcoal, will be analyzed for each vapor collection sample. The list of the specific compounds follows. The methods listed above are the NIOSH methods which include this list of analytes. The sampling and analysis conditions are the same for the NIOSH methods, so for the purposes of this analysis, the analytes will all be determined using the same analytical method.

Analytes:

Acetone
Chlorobenzene
Chloroform
1,1-Dichloroethane
1,1-Dichloroethylene (Vinylidene chloride)
Ethyl Benzene
Methyl Ethyl Ketone
Methyl Isobutyl Ketone
Methylene Chloride (Dichloromethane)
Tetrachloroethylene (Perchloroethylene)
Toluene
1,1,1-Trichloroethane (Methyl Chloroform)
1,1,2-Trichloroethane
Trichloroethylene
Xylenes (ortho, meta, and para isomers)

Section No. 9 Revision No. 1 March 16, 1992 Page 4 of 20

Apparatus and Reagents:

- 1. Hewlett-Packard 5890 series Gas Chromatograph with FID detection (or equivalent). The instrument must be configured with capillary column capability. Dual auto samplers and detectors may be used to allow for dual column operations. The gas chromatographic system will be equipped with an integrator/data system which allows for calibration of the instrument and quantification of the chromatograms using external standards.
- 2. Fused silica capillary gas chromatographic column: 30 meters long with 0.32mm internal diameter with 1.0um SPB-5 bonded phase (or equivalent).
- 3. Fused silica capillary gas chromatographic column: 30 meters long with 0.32mm internal diameter with 1.0um DB-WAX bonded phase (or equivalent).
- 4. Carbon Disulfide: glass distilled, HPLC/Spectral grade (or equivalent). This material has been shown to contain benzene. A further cleanup of the solvent is required if benzene is determined. The benzene is removed using 13X molecular sieves. Approximately 50 grams of the molecular sieves are added to a 2.5 liter bottle of carbon disulfide and the bottle mixed by swirling. The materials are equilibrated overnight. The molecular sieves are removed by filtering the carbon disulfide. The molecular sieves are $\mbox{discarde}\bar{\mbox{d}}$ after allowing the carbon disulfide to evaporate in a hood. The carbon disulfide is returned to the original bottle and a second quantity of molecular sieves added. process is repeated a total of five times. The benzene concentration must be below 1 ug/mL in the cleaned up solvent.
- 5. Reagent grade standards for all analytes listed above. The neat materials must have a listed purity of greater than 95%.
- 6. Desorption vials: 4 mL vials with screw top lids with PTFE or other material which is impervious to carbon disulfide.
- 7. Autosampler vials: 1.5 mL vials to fit autosampler on gas chromatograph. Either a screw top or crimp closure can be used. The septum must be PTFE lined and must not contribute any components to the solvent blank used for the analysis.
- 8. Glass sampling tubes of approximately 4 to 5 cm in length (4 mm ID x 6 mm OD) which are packed with a 100 mg from section and a 50 mg back section of 20/40 mesh activated coconut shell charcoal separated by a 2 mm portion of urethane foam. A plug of silylated glass wool is placed in front of the absorbing section and a 3 mm portion of urethane foam is placed behind the back section of charcoal. SKC part # 226-01 or equivalent.

Section No. 9 Revision No. 1 March 16, 1992 Page 5 of 20

Chromatographic Conditions:

Injector temperature: 250 C
Detector temperature: 320 C
Detector Range: 2^5
Detector Attenuation: 2^-2
Flow Rate (approximate): 2 mL/min
Purge flow: 3 mL/min
Splitter flow: 90 mL/min

Temperature Program:

Initial 35 C - 4 min.
Program rate 9 C/min
Final 200 C - 1 min
Inlet Pressure: 3.5 psi
Injection volume: 2 uL (split)

Instrument Maintenance:

Routine instrument maintenance will be carried out on a regular basis, at minimum once a week. This will include inspection and replacement of the septum, injector syringe, and other consumable items. This routine maintenance will be recorded in the instrument log book. Clean up, replacement of columns and other non-routine maintenance will also be recorded in the instrument log.

Safety Precautions:

Carbon disulfide is extremely flammable and considered toxic. The OSHA TLV (an eight hour TWA) is 10 ppm. Both inhalation and skin exposure should be avoided. Large quantities of the solvent should be handled in a hood.

Many of the analytes also are inhalation hazards.

1,1-Dichloroethylene, 1,1,2-trichloroethane,
trichloroethylene, chloroform, and tetrachloroethylene have
been tentatively classified as known or suspected human or
mammalian carcinogens. The handling of the neat materials
should be performed with gloves and be in a hood.

Preparation of calibration standards:

Stock calibration standards: Weigh approximately 0.1 g for nonchlorinated compounds and 0.3 g for chlorinated compounds into a 10 mL volumetric containing approximately 2 mL of carbon disulfide. The standards are prepared in the order listed below. This order is roughly the reverse of the vapor pressure to prevent excessive evaporation of the more volatile compounds. After all the compounds are weighed into the volumetric, dilute to 10 mL with carbon disulfide.

Section No. 9 Revision No. 1 March 16, 1992 Page 6 of 20

Chlorobenzene
Tetrachloroethylene
Xylenes
Ethyl Benzene
Trichloroethylene
Toluene
1,1,2-Trichloroethane
1,1,1-Trichloroethane
Methyl Isobutyl Ketone
Methyl Ethyl Ketone
Chloroform
Acetone
Methylene Chloride
1,1-Dichloroethane
1,1-Dichloroethylene

Working calibration standards are prepared at the following dilutions in carbon disulfide:

DF	20	0.5 mL stock diluted to 10 mL
DF	40	0.25 mL stock diluted to 10 mL
DF	100	0.1 mL stock diluted to 10 mL
DF	400	0.5 mL of DF 20 solution diluted to 10 mL
DF	2000	0.25 mL of DF 20 solution diluted to 25 mL
DF	4000	1.0 mL of DF 400 solution diluted to 10 mL

All measurement of the stock and working solutions' volume prior to dilution should be using pipets or syringes. After all the analytes have been added, add carbon disulfide to make the total volume 10 mL. After the stock and working standards are prepared they are transferred to a glass vial with a PTFE lined lid and stored in a freezer. The stock standards can be stored for up to 30 days under freezer conditions, the working standards can be stored for up to 1 week in the freezer before being replaced.

Preparation of a surrogate standard:

Weigh 0.15 g of n-butanol into a 10 mL volumetric containing 2 mL of carbon disulfide. After the neat surrogate material is weighed into the volumetric, dilute to 10 mL with carbon disulfide. Transfer the solution to a vial with screw cap and PTFE lined lid. Stored in a freezer the solution is stable for 1 week.

Section No. 9 Revision No. 1 March 16, 1992 Page 7 of 20

Preparation of a spiking standard:

Prepare a stock spiking solution as follows: Weigh approximately 0.1 g of each of the nonchlorinated and 0.3 g of the chlorinated compounds into a 10 mL volumetric and dilute to 10 mL. This produces a stock solution of 10 to 30 mg/mL. This will be a separate solution than the stock calibration standard. The same storage conditions and times apply as for the calibration standard.

Procedure:

- 1. Samples upon receipt are placed into refrigerated storage until analysis. Analysis will occur within seven days from receipt.
- 2. Intact charcoal tubes are scored with a file and broken open at each end. The glass wool plug is removed with a fine wire hook. The charcoal from each section of the first tube is combined, added to a 4 mL vial and identified as the "front". Similarly, the charcoal from the second tube in line is combined, added to a 4 mL vial and identified as the "back".
- 3. To each vial containing charcoal, carbon disulfide is accurately transferred with a syringe. For each 100 mg/50 mg tube analyzed as one section, use three mL of carbon disulfide for the desorption. Add the surrogate standard solution at this point, 20 microliters for the 3.0 mL volume of carbon disulfide. Immediately cap the vial and shake the vial for at least 30 seconds. Desorption should be complete after 30 to 45 minutes. The vial should be mixed by shaking at least two times during this period. Allow the charcoal to settle to the bottom of the vial before removing the solvent.
- 4. Transfer between 1.0 and 1.5 mL of the desorption solvent from each vial used for desorption to a GC autosampler vial. Be sure to identify the "front " and "back" sections on the GC autosampler vials. Cap the vials immediately.
- 5. Prepare the working calibration standards in GC autoinjector vials, adding approximately 1.0 mL of the working solutions to a vial. Five levels of standards are used (DF 20, DF 40, DF 100, DF 400, DF 2000). A DF 4000 standard is used to determine the quantification limit. A complete set of standards are analyzed each sample batch, and a check standard made up of the mid level (DF 100) standard is analyzed at least once for every ten samples.

Section No. 9 Revision No. 1 March 16, 1992 Page 8 of 20

- 6. Prepare two spiked tubes as described below:
 - A. Score intact charcoal tubes with a file and break open at each end. Remove the glass wool plug with a fine wire hook. The charcoal from each section of a single tube is removed as described above, combined, and added to a 4 mL vial.
 - B. To each vial containing charcoal, transfer carbon disulfide with a syringe measuring the volume exactly. Three mL of carbon disulfide is used for desorption of all the carbon in the tube. Add the surrogate standard solution at this point, 20 microliters to the 3.0 mL volume of carbon disulfide followed by 10 uL of the spiking stock solution.

Immediately cap the vial and shake the vial for at least 30 seconds. Desorption is complete after 30 to 45 minutes. The vial should be mixed by shaking at least two times during this period. Allow the charcoal to settle to the bottom of the vial before removing the solvent.

- C. Transfer between 1.0 and 1.5 mL of the solvent from each vial used for desorption to a GC autosampler vial. Cap the vials immediately.
- 7. Prepare a media blank by desorbing a sealed charcoal tube which is from the same lot as the tubes used for the matrix spike/matrix spike duplicate analysis. The procedure is the same as that identified in sections 2-4. This result will be used to calculate the recovery of analyte from the spiked tubes.
- 8. Prepare a solvent blank by adding 1.5 mL of the carbon disulfide plus 10 uL of the surrogate standard to a GC autosampler vial. Cap the vial immediately.
- 9. Analyze the calibration standards, a solvent blank (with surrogate standard added), the DF 4000 quantification limit standard, the check standard (roughly one every 10th sample) and the spiked tubes as described above along with the samples for each batch of samples (up to 20 samples). A MDL study is included in Table 3. The expected detection limit is listed in a Table 1.
- 10. After the samples have been analyzed, review the retention times for all calibration and check standards. If the retention times are within 0.04 minutes or 0.2% of retention time (whichever is larger), the initial retention times can be used to identify the components. If the retention times vary more than this amount the retention times should be updated based on each check standard.

Section No. 9 Revision No. 1 March 16, 1992 Page 9 of 20

The SPB-5 capillary column is used as the primary column for all analytes except for the xylenes. The DB-Wax column is used as the primary column for the xylenes. The primary column is used for quantitation of the analyte, however if interferences are present in the chromatogram, quantitation of the analyte may be based on the confirmatory column. The retention times for any component should match the retention times for standards on both columns as listed above.

- 11. Review the chromatograms, any samples which have analyte responses greater than the highest level standard must be diluted and reanalyzed. These should be analyzed in the same analytical batch if possible.
- 12. Calibrate the system based on the peak height of the five levels of standards. Calculate an average response factor based on the amount in the standards (in ug/mL) per peak height unit. The average response factor is used to calculate the concentration as long as the response factor varies less than 25% from the average. If the variation is greater than 25%, a calculation based on the response factor of the standard which is closest to the peak height of the analyte is required.
- 13. Calculate the recovery from the spiked tubes. The recovery must be within 25% of the expected value. The relative percent difference between the two results for the spiked tubes must also be less than 15%.

Calculations:

The quantity of analyte per tube is calculated using the following formula:

```
mg analyte/tube = [(A)xCFxDxDV/1000]
```

where A = sample peak height

CF = calibration factor {amount (ug/mL)/peak height}

D = dilution factor

DV = desorption volume in mL

The quantity of analyte in the vapor phase is calculated using the following formula:

ppm (v/v) analyte = [(M/V)x(24.45/MW)x((T+273)/298)x(760/P)]

where

M = mg analyte/tube

V = volume collected in cubic meters

T = temperature in C

P = pressure in mm Hg

MW = molecular weight of the analyte

Section No. 9 Revision No. 1 March 16, 1992 Page 10 of 20

The mg analyte per tube is the sum of the levels on both tubes. If components are found on the back tube, calculate the % breakthrough using this formula:

% Breakthrough = $B_{mq}/F_{mg} \times 100$ %

Where $B_{mg} = mg$ of analyte found on the back tube and $F_{mq} = mg$ of analyte found on the front tube.

If this is greater than 25%, the quantity reported should be qualified on the analytical report, indicating that breakthrough of the analyte has occurred and the % breakthrough.

Quality Control:

Spikes: Two unopened tubes will be spiked as a spike and spike duplicate with the compounds of interest and analyzed with each analytical batch of 20 samples. The recovery must be within 25% of the expected and the relative percent difference (RPD) between the spiked tubes must be less than 15%. The spiking levels are approximately 0.1 mg (nonchlorinated compounds) and 0.3 mg (chlorinated compounds) per tube.

The approximate concentration in the final desorption solution and the mass of spike added to the tube is listed below.

O	Solution Concentration	Spike Added
Compound	(mg/mL)	(mg)
Acetone	.033	0.1
Chlorobenzene	.100	0.3
Chloroform	.100	0.3
1,1-Dichloroethane	.100	0.3
1,1-Dichloroethene	.100	0.3
Ethyl benzene	.033	0.1
Methyl ethyl ketone	.033	0.1
Methyl isobutyl ketone	.033	0.1
Methylene chloride	.100	0.3
Tetrachloroethylene	.100	0.3
Toluene	.033	0.1
1,1,1-Trichloroethane	.100	0.3
1,1,2-Trichloroethane	.100	0.3
Trichloroethylene	.100	0.3
Xylenes (sum of isomers	.033	0.1

Section No. 9 Revision No. 1 March 16, 1992 Page 11 of 20

No data base of recovery information is available to evaluate the recovery or RPD measurements. Replicate analysis of spiked tubes may be performed to check for analyst error if the spikes can be performed in the same analytical batch. If the recovery or RPD values on the repeat spikes are outside these windows the data will be flagged with a comment on the analytical report.

Blanks: At least one solvent blank will be analyzed per batch of samples. No analyte of interest may be present in the solvent blank at a level equal to the DF 4000 standard. If analytes are found above this level the data will be flagged with a comment on the analytical report.

Surrogate standard: A surrogate standard, n-butanol, will be added to each sample spike and blank at approximately 0.1 mg/mL of desorption solvent. The surrogate recovery should be within 25% of the expected value. If problems with the surrogate standards are noted but the spikes of tubes give acceptable results, an alternate surrogate material may be used.

Check standard: The mid level (DF100) standard will be used as a check standard. This will be analyzed after every tenth sample, at least one check standard will be analyzed per batch. For a batch of 11 to 20 samples, two check standards will be analyzed. This standard must be within 15 % of the expected value. If it is not, repeat the analysis of the check standard or a fresh supply of the standard. If that is within the acceptance range report the new standard. If that is still outside the acceptance range, repeat the calibration standards and all samples since the last check standard which was within the acceptance range. If it is not possible to repeat the analysis the data must be flagged with a comment on the report.

Section No. 9 Revision No. 1 March 16, 1992 Page 12 of 20

Quality Control Samples used to assess the analyses:

Media blanks	(MB)
Solvent blanks	(SB)
Field blanks	(FB)
Travel blanks	(TB)
Spiked tube	(MS)
Duplicate spiked tube	(MSD)

The solvent blank is used to determine the possible contamination of samples while in the laboratory. The solvent blank is generated in the laboratory.

The field blank and travel blank are used to determine the possible contamination of samples while in the field. These blanks are generated in the field.

The media blank is used to correct the spiked tube and duplicate spiked tube for any contaminants found in the media. The media blank is generated in the laboratory from the same batch of media as was used in the field.

The spiked tube and duplicate spiked tube are used to assess the precision and accuracy of the analysis. The spiked tube and duplicate spiked tube are generated in the laboratory.

Table 1

Limit of quantitation for soil vapor analyses Selected VOCs on Charcoal tubes: Limit of Quantitation (LOQ)

FLOWRATE: TIME:	.2 (L/min) 50 (min)	Atm Pressure:	760 mmHg
VOLUME: TEMP (C):	10 (Liters) 25	Desorb Vol:	3 mL
Compound	Log	LOQ mg/m3	Analyte LOQ (mg/tube)
Acetone	.35	.82	.0082
Chlorobenzene	.29	1.36	.0136
Chloroform	.46	2.24	.0224
1,1-Dichloroethane	.32	1.28	.0128
1,1-Dichloroethene	.30	1.18	.0118
Ethyl benzene	.13	.56	.0056
Methyl ethyl ketone	.30	.88	.0088
Methyl isobutyl ketone	.22	.90	.0090
Methylene chloride	.63	2.20	.0220
Tetrachloroethylene	.61	4.16	.0416
Toluene	.21	.78	.0078
1,1,1-Trichloroethane	.40	2.20	.0220
1,1,2-Trichloroethane	.40	2.16	.0216
Trichloroethylene	. 45	2.42	.0242
Xylenes	.20	.88	.0088

LOQ is based on an MDL study for the analysis.

Phenol on XAD-7 tube: Limit of Quantitation (LOQ)

FLOWRATE: TIME:	(L/min) (min)	Atm. Pressure:	760 mmHg
VOLUME: TEMP (C):	(Liters)	Desorb Vol:	2 mL
Compound	 LOQ ppm	LOQ mg/m3	Analyte LOQ (mg/tube)
Phenol	.20	.76	.0076

LOQ is based on an MDL study for the analysis.

Table 2

Dynamic Range: Selected VOCs on charcoal tubes

Compound	Lower Limit mg/tube	Upper Limit mg/tube	Lower Limit ppm	Upper Limit ppm
Acetone	.015	1.50	.632	63.2
Chlorobenzene	.045	4.50	.974	97.4
Chloroform	.045	4.50	.925	92.5
1,1-Dichloroethane	.045	4.50	1.112	111.2
1,1-Dichloroethene	.045	4.50	1.135	113.5
Ethyl benzene	.015	1.50	.346	34.6
Methyl ethyl ketone	.015	1.50	.509	50.9
Methyl isobutyl keton	■ .015	1.50	.367	36.7
Methylene chloride	.045	4.50	1.294	129.4
Tetrachloroethylene	.045	4.50	.663	66.3
Toluene	.015	1.50	.399	39.9
1,1,1-Trichloroethane	.045	4.50	.827	82.7
1,1,2-Trichloroethane		4.50	.827	82.7
Trichloroethylene	.045	4.50	.840	84.0
Xylenes	.015	1.50	.346	34.6

Dynamic Range: Phenol on XAD-7 tube

Compound	Lower Limit mg/tube	Upper Limit mg/tube	Lower Limit	Upper Limit ppm
Phenol	.008	.76	.197	19.7

If levels greater than the upper limit for the analysis are detected, the tube extract will be diluted to bring the concentration within the analytical dynamic range.

Section No. 9 Revision No. 1 March 16, 1992 Page 15 of 20

Table 3

MDL Calculation: Selected Organics on Charcoal tubes

Analyte	Std. Conc. (ug/mL)	# Reps	SD (n-1)	MDL (ug/tube)	PQL (ug/tube)	
	<u>-</u>					
Acetone	5.08	7	.0742	.413	4.1	
1,1-Dichloroethylene	10.06	7	.1059	. 589	5.9	
1,1-Dichloroethane	10.14	7	.1146	.638	6.4	
Methyl Ethyl Ketone	5.17	7	.0791	. 440	4.4	
Chloroform	10.33	7	.2007	1.117	11.2	
1,1,1-Trichloroethane	10.03	7	.1993	1.109	11.1	
Carbon Tetrachloride*	10.33	7	.2480	1.380	13.8	
Trichloroethylene	10.02	7	.3080	1.714	17.1	
Methyl Isobutyl Ketone	5.31	7	.0815	.453	4.5	
Toluene	5.38	7	.0704	.392	3.9	
1,1,2-Trichloroethane	10.29	7	.1940	1.080	10.8	
Tetrachloroethylene	10.22	7	.3746	2.084	20.8	
Chlorobenzene	10.20	7	.1227	. 683	6.8	
Ethyl benzene	5.38	7	.0501	.279	2.8	
p-Xylene	1.74	7	.0573	.319	3.2	
m-Xylene	1.70	7	.0484	.269	2.7	
o-Xylene	1.86	7	.0782	.435	4.4	

^{*} Carbon Tetrachloride studied rather than methylene chloride. The MDL for these two compounds is similar.

Analysis of Phenol on XAD-7 Tube for ECC Site - Soil Vapor Analysis

References:

OSHA Method 32

Principle:

Organic vapors in soil gas are collected on XAD-7 tubes by passing the gas through the tubes at a controlled rate for a specified period of time. Phenol is removed from tube by desorbing the tube with methanol and analyzing the solvent by high performance liquid chromatography using a ultraviolet (UV) detector at 218 nm. Knowing the exact volume of soil gas passed through the tube and the mass of the organic compound sorbed on the tube, the concentration of the compound in the soil gas can be calculated.

Scope:

This method is applicable to the analysis of phenol sorbed on XAD-7. One 100 mg/50 mg XAD-7 tube will be analyzed for each vapor collection sample.

Apparatus and Reagents:

- 1. A Shimadzu high performance liquid chromatograph (HPLC) equipped with a sample injector, reverse-phase HPLC column (see item 2), variable wavelength detector, integrator/data system. The data system allows for calibration of the instrument and quantification of the chromatograms using external standards.
- 2. Reverse phase stainless steel column (25 cm long X 4.6 mm ID), HPLC column packed with Whatman 5 ODS 3 packing material (or equivalent).
- 3. HPLC grade methanol.
- 4. Deionized water.
- 5. Reagent grade phosphoric acid (H3PO4).
- 6. Reagent grade standard of phenol from Chem Service or other supplier which indicates the purity.
- 7. Glass sampling tubes of approximately 4 to 5 cm in length (4mm ID x 6mm OD) which are packed with 100 mg front section, and a 50 mg back section of 15/50 mesh XAD-7 resin. Small silanized glass wool plugs are placed in the ends and in the middle between the sections of resin. SKC part number 226-30-12-07 or equivalent.

Section No. 9 Revision No. 1 March 16, 1992 Page 17 of 20

Chromatographic Conditions:

Injector volume: 25 uL Detector wavelength: 218 nm

Mobile Phase: 59/41 (v/v) methanol/water

mixture with 0.1% H3PO4.

Flow Rate: 1 mL/min.

Instrument Maintenance:

Routine instrument maintenance will be carried out on a regular basis, as described in SOP MC-FC-009.

Safety Precautions:

Methanol is flammable and must be handled with care. Skin exposure should be avoided. Large quantities of the solvent should be handled in a hood.

Preparation of calibration standards:

Stock calibration standards: Weigh approximately 0.12 g of phenol into a 25 mL volumetric and dilute to volume with methanol. Working calibration standards are prepared at the following dilutions in methanol:

DF	200	1.0 m	L stock	diluted	to	200 mL	(24	ug/L)
DF	100	1.0 m	L stock	diluted	to	100 mL	(48	ug/L)
DF	50	1.0 m	L stock	diluted	to	50 mL	(96	ug/L)
DF	20	1.0 m	L stock	diluted	to	25 mL	(192	ug/L)
DF	12.5	2.0 m	L stock	diluted	to	25 mL	(384	ug/L)

A quantification limit standard (DF 10000) will be prepared by diluting 1 mL of the DF 100 standard into 100 mL with methanol. All measurement of the of the stock and working solutions' volume prior to dilution should be using pipettes. Transfer the standard solutions in brown bottles with Teflonlined caps for storage in the refrigerator for 15 days.

Section No. 9 Revision No. 1 March 16, 1992 Page 18 of 20

Calibration:

The standards listed above will be injected at a minimum in duplicate throughout the analytical run. A standard will be injected at least after every fourth sample injection. A linear least squares fit of the calibration standards data will be used to calibrate the chromatographic system. The correlation coefficient must be greater than or equal to 0.995. The samples will be calculated with the new calibration to determine the phenol concentration. The percent difference in response between two standard injections of the same standard concentration should be < 10%. The RSD between three or more standard injections of the same standard concentration should be < 5%.

Preparation of a spiking standard:

Stock spiking solution: Weigh approximately 0.12 g of phenol into a 25 mL volumetric and dilute to volume with methanol. This solution is approximately 4.8 mg/mL. This will be a separate solution than the stock calibration standard. The same storage conditions and times apply as for the calibration standard.

Procedure:

- 1. Samples upon receipt are placed into refrigerated storage until analysis. Analysis will occur within forteen days from receipt.
- 2. Transfer the front glass wool and sorbent section of the sampling tube to a 4-mL vial. Label as front section. Place the remaining backup section including both glass wool plugs into a separate 4-mL vial. Label as back section.
- 3. Add 2 mL of methanol to each vial. Immediately cap the vial, and shake it on a mechanical shaker for 15 minutes.
- 4. Transfer between 1.0 and 1.5 mL of the desorption solvent from each vial used for desorption to a HPLC autosampler vial. Be sure to identify the "front " and "back" sections on the HPLC autosampler vials. Cap the vials immediately.
- 5. Transfer the working calibration standards in HPLC autosampler vials. Five levels of standards are used. Two vials of each standard (at a minimum) are injected with each batch. A standard is injected at least after every fourth sample injection.

Section No. 9 Revision No. 1 March 16, 1992 Page 19 of 20

6. Prepare two spiked tubes as described below:

Add a 4 uL aliquot of the spiking stock to each of two clean XAD-7 sampling tubes. This results in a spike level of approximately 19 ug of phenol or 9.6 ug/mL in the desorption solvent. Desorb the spiked samples starting with step 2 of the analytical procedure.

- 7. Prepare a media blank by desorbing a XAD-7 sampling tube which is from the same lot as the tubes used for the matrix spike/matrix spike duplicate analysis. The procedure is the same as that identified in sections 2-4. This result will be used to calculate the recovery of analyte from the spiked tubes.
- 8. Prepare a solvent blank by adding 1.5 mL of the methanol to an HPLC autosampler vial. Cap the vial immediately.
- 9. Analyze the calibration standards, a solvent blank, the DF 10000 quantification limit standard, and the spiked tubes as described above along with the samples for each batch of samples (up to 20 samples).
- 10. Review the chromatograms, any samples which have analyte responses greater than the highest level standard must be diluted and reanalyzed. These should be analyzed in the same analytical batch if possible.
- 11. Calibrate the system based on the peak responses of the five levels of standards. Perform a linear least squares fit of the standards data to determine a line to calibrate the chromatographic system. The correlation coefficient must be greater than or equal to 0.995. The samples will be calculated with the new calibration to determine the phenol concentration.
- 12. Calculate the recovery from the spiked tubes. The recovery must be within 25% of the expected value. The relative percent difference between the two results for the spiked tubes must also be less than 15%.

Calculations:

The quantity of analyte per tube is calculated using the least squares fit of the standards data as performed on the Shimadzu integrator. The mg/tube result of the sample is calculated by the integrator following the linear equation of:

$$y = Ax + B$$

(The volume used for desorbing the tube is entered into integrator by the use of dilution factor.)

Section No. 9 Revision No. 1 March 16, 1992 Page 20 of 20

The quantity of analyte in the vapor phase is calculated using the following formula:

ppm (v/v) analyte = [(M/V)x22.45x((T+273)/298)x(760/P)]94.11

where M = mg analyte/tube

V = volume collected in cubic meter

T = temperature in C
P = pressure in mm Hg

94.11 = MW of Phenol

The mg analyte per tube is the sum of the levels in both sections of the tube. If components are found on the back section, calculate the % breakthrough using this formula:

 $$ Breakthrough = B_{mq}/F_{mq} \times 100$$

Where $B_{mg} = mg$ of analyte found on the back section $F_{mg} = mg$ of analyte found on the front section.

If this is greater than 25%, the quantity reported should be qualified on the analytical report, indicating that breakthrough of the analyte has occurred and the % breakthrough.

Quality Control:

Spikes: Two unopened tubes will be spiked with a 4 uL aliquot of the stock spiking solution and analyzed with each analytical batch of 20 samples. The recovery must be within 25% of the expected and the relative percent difference (RPD) must be less than 15%. The spiking level is approximately 19 ug per tube or 9.6 ug/mL in the desorption solvent. No data base of recovery information is available to evaluate the recovery or RPD measurements. Replicate analysis of spiked tubes may be performed to check for analyst error if the spikes can be performed in the same analytical batch. If the recovery or RPD values on the repeat spikes are outside these windows the data will be flagged with a comment on the analytical report.

Blanks: At least one solvent blank will be analyzed per batch of samples. No analyte of interest may be present in the solvent blank at a level equal to or above the DF 10000 standard. If analytes are found above this level the data will be flagged with a comment on the analytical report.

Section No. 10 Revision No. Date: 11/14/91 Page 1 of 5

10. Data Reduction, Validation and Reporting

Raw analytical data generated in the laboratories is collected on printouts from the instruments and associated data system or manually in bound notebooks. Analysts review data as it is generated to determine that the instruments are performing within specifications. This review includes calibration checks, surrogate recoveries, blank checks, retention time reproducibility, and other QC checks described in Section No. 11. If any problems are noted during the analytical run, corrective action is taken and documented.

Each analytical run is reviewed by a chemist for completeness prior to interpretation and data reduction. The following calculations are used to reduce raw data to reportable results.

<u>Volatile Organics in Air</u> - The quantity of analyte per tube is calculated using the following formula:

mg analyte/tube = [(A)xCFxDxDV/1000]

Where A = sample peak height

CF = calibration factor [amount (ug/ml)/peak height]

D = dilution factor

DV = desorption volume in ml

The quantity of analyte in the vapor phase is calculated using the following formula:

ppm (v/v) analyte = [(M/V)x(24.45/MW)x(T+273)/298)x(760/P)]

Where M = mg analyte/tube

*

V = volume collected in cubic meters

T = temperature in C

P = pressure in mm Hg

MW = molecular weight of the analyte

Section No. 10 Revision No. Date: 11/14/91 Page 2 of 5

<u>Phenol in Air</u> - Include in the calculations the concentration of the analyte found on the front and backup sections of a sampling tube. Express results in mg/m^3 using the following equations:

ug/ml (total) = ug/ml (front section) + ug/ml (backup section) $mg/m^3 = (ug/ml(total))$ (2 ml desorption) / air volume in liters

To convert to ppm at 760 mm and 25°C:

 $ppm = (mq/m^3)(24.45)/(MW \text{ of analyte})$

24.45 is the molar volume of an ideal gas at 760 mm Hg, 25°C.

The principle criteria used to validate data will be the acceptance criteria described in Section No. 11. Following interpretation and data reduction by an analyst, data is transferred to the laboratory sample management system either by direct data upload from the analytical data system or manually. The data is reviewed by the Group Leader or another analyst and verified on the sample management system. The person performing the verification step reviews all data including quality control information prior to verifying the data. If data package deliverables have been requested, the laboratory will complete the appropriate forms (see Appendix A) summarizing the quality control information, and transfer copies of all raw data (instrument print-outs, spectra, chromatograms, laboratory notebooks, etc.) to the Data Packages Group. This group will combine the information from the various analytical groups and the analytical reports from the laboratory sample management system into

Section No. 10 Revision No. Date: 11/14/91 Page 3 of 5

one package. This package is reviewed by the Quality Assurance Department for conformance with SOP's and to ensure that all QC goals have been met. Any analytical problems are discussed in the case narrative, which is also included with the data package deliverables.

The validation of the data by the Quality Assurance Department includes spot checking raw data versus the final report, checking that all pertinent raw data is included and does refer to the samples analyzed, review of all QC results for conformance with the method, and review of the case narrative for description of any unusual occurrences during analysis. This validation is performed using techniques similar to those used by the Sample Management Office for the USEPA's Contract Laboratory Program. The validation performed by the laboratory does not address useability of the data, which usually requires some knowledge of the site. The laboratory will make every attempt to meet the requirements of this QAPP, thus reducing the need to assess useability of the data:

The laboratory sample management system is programmed to accept and track the results of quality control samples including blanks, surrogates, recoveries, duplicates, controls, and reference materials. The computer is programmed with the acceptance criteria for each type of QC sample and will display an out-of-spec message if the data is not within specifications. All data outside of specifications appears on a report to the Quality Assurance Department on the next working day. These are reviewed by the Quality Assurance Department for severity of the problems and trends in the data. The reports are then sent to the analytical groups for the purpose of

Section No. 10 Revision No. Date: 11/14/91 Page 4 of 5

documenting the corrective action taken. The sample management system also produces control charts and has searching capabilities to aid in data review. The flow of data from the time the samples enter the laboratory until the data is reported are summarized in Table 10-1. data recorded manually will be collected in bound notebooks. All entries will be in ink, with no erasures or white-out being permitted. Any changes in data will be made using a single line to avoid obliteration of the original entry and will be dated and signed. Any data resulting from instrument printouts will be dated and will contain the signature and/or identification of the analyst responsible for its generation. After copies of the data are incorporated into the data package deliverables, the originals will be stored in locked archives at the laboratory for a period of ten years.

Section No. 10 Revision No. Date: 11/14/91 Page 5 of 5

Table 10-1

SAMPLE AND DATA ROUTING AT LANCASTER LABORATORIES, INC.

Action	Personnel Involved
Sample received at LLI	Sample Administration
Sample is entered onto sample management system (lab ID number assigned, analyses scheduled, chain-of-custody started, storage location assigned)	Sample Administration
Sample stored in assigned location (refrigerator, freezer, etc.)	Sample Administration
Acknowledgement sent to client	Sample Administration
Removed from storage for analysis; tube is desorbed; extracts retained in the laboratory	Technical Personnel
Analysis is performed according to selected analytical method; raw data recorded in notebook and transferred to computer by chemist or technician*	Technical Personnel
Computer performs calculations as programmed according to methods	Data Processing
. Chemist or supervisor verifies raw data	Technical Personnel
Data package deliverables are assembled	Data Package Group
Data packages are reviewed prior to mailing	Quality Assurance Dept. Laboratory Management

^{*}Analyses requiring the chemist's interpretation may involve manual data reduction prior to entry onto the computer.

Section No. 11 Revision No. Date: 11/14/91 Page 1 of 4

11. Internal Quality Control Checks

The particular types and frequencies of quality control checks analyzed with each sample are defined in the methods in Section 9. The quality control checks routinely performed during sample analysis include surrogates, matrix spikes, and blanks.

<u>Surrogates</u> (used for organic analysis only) - Each sample, matrix spike, matrix spike duplicate, and blank are spiked with a surrogate compound during desorption in order to monitor desorption and analysis. Surrogates are used to evaluate analytical efficiency by measuring recovery.

Matrix Spikes - A matrix (blank sorbant tube) is spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

<u>Duplicates</u> (matrix spike duplicate) - A second blank sorbant tube is analyzed at the same time as the original sample in order to determine the precision of the method. Recovery of the original compared to the duplicate is expressed as relative percent differences (RPD).

Section No. 11 Revision No. Date: 11/14/91 Page 2 of 4

Blanks (Media, Method) - Blanks are unopened sorbant tubes from the same batch as those used in the field. They are opened in the laboratory and treated with the same reagents and surrogate standards as samples and carried through the entire analytical procedure.

The charts that follow show the types and frequency of QC performed, along with the acceptance limits.

Section No. 11 Revision No. Date: 11/14/91 Page 3 of 4

QC Charts

Type	Acceptance Limits	Frequency
- VOLATILES BY GC:		
Surrogate:		
n-butanol	85-125	Each sample, MS, MSD, and blank.
- Matrix Spike:		
Acetone Chlorobenzene Chloroform 1,1-Dichloroethane 1,1-Dichloroethene Ethyl benzene Methyl ethyl ketone Methyl isobutyl ketone Methylene chloride Tetrachloroethylene Toluene 1,1,1-Trichloroethane 1,1,2-Trichloroethane Trichloroethylene Xylenes (sum of isomers)	75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125	Each group (<20) of samples.
Matrix Spike Duplicate (RPD):		
- Acetone Chlorobenzene Chloroform 1,1-Dichloroethane 1,1-Dichloroethene Ethyl benzene Methyl ethyl ketone - Methyl isobutyl ketone Methylene chloride Tetrachloroethylene Toluene 1,1,1-Trichloroethane 1,1,2-Trichloroethane Trichloroethylene Xylenes (sum of isomers)	15 15 15 15 15 15 15 15 15 15 15 15	Each group (<20) of samples.
Blank _	<pre>< level of DF4000 std</pre>	Each group (\leq 20) of samples.

Section No. 11 Revision No. Date: 11/14/91 Page 4 of 4

QC Charts (Continued)

-	Type	Acceptance Limits	Frequency
	PHENOLS BY HPLC:		
_	Matrix Spike:		
	Phenol	75-125	Each group (≤ 20) of samples.
	Matrix Spike Duplicate (RPD):		
_	Phenol	15	Each group (≤ 20) of samples.
	Blank	<u><</u> LOQ	Each group (\leq 20) of samples.

Section No. 12 Revision No. Date: 11/14/91 Page 1 of 15

12. Performance and System Audits

System audits are conducted on each department at Lancaster Laboratories, Inc. (LLI) by members of the Quality Assurance Department. The audits include checks on methodology, reagent preparation, equipment calibration and maintenance, quality control results, and training of personnel. The results of the audits and corrective action, where necessary are communicated to laboratory personnel and management by means of a written report. Audits by outside organizations including clients, regulatory personnel and the USEPA are permitted by arrangement with the Quality Assurance Department.

On a monthly basis, the Quality Assurance Department reviews summaries of the quality control data entered onto the computerized sample management system by analysts. Control charts and statistics are reviewed for trends which may indicate problems with the analytical data. In this way, small problems are identified before they have any significant impact on laboratory results.

Performance audits consist of both intralaboratory and interlaboratory check samples. Blind samples containing known amounts of target analytes are prepared by the Quality Assurance Department and submitted to the laboratories under fictitious client names. In addition, QC samples from EMSL-Cinncinnati are analyzed quarterly to assess laboratory accuracy. LLI also participates in a number of interlaboratory performance evaluation studies which involve analysis of samples with concentrations of analytes that are known to the sponsoring organization, but unknown to the laboratory. Inorganics,

Section No. 12 Revision No. Date: 11/14/91 Page 2 of 15

pesticide/herbicides, trihalomethanes, volatile organic compounds, semivolatile organic compounds and traditional wet chemistry analyses are analyzed by LLI for studies conducted by the USEPA and the New York Department of Health. LLI is a contractor to the USEPA under the Contract Laboratory Program which provides laboratory analysis in support of the Superfund program. Part of maintaining this contract includes analysis of quarterly blind samples. Interlaboratory check samples are also provided through the American Industrial Hygiene Association accreditation program. The Proficiency Analytical Testing Program (PAT Program) submits samples to the laboratory quarterly. Charcoal tubes with quantities of organics known to the sponsoring organization are trapped on charcoal tubes at levels unknown to the laboratory. Representative results from some of these PAT Program rounds are attached to this section. Representative results from some of these studies are attached to this section.

PERFORMANCE EVALUATION REPORT

DATE: 12/21/90

FATER POLLUTION STUDY BURBER RP025

LABORATORY: PAG	707	
-----------------	-----	--

ANALYTES	SANGLE . REEMUR				WARNING LIMITS	PERFORMANCE EVALUATION
TRACE ME	TALS IN MICE	ROGRAMS	PER LIT	er:		
ALUMINUM	1 2			1150- 1690 20.6- 35.7	1220- 1520 23.8- 77.4	ACCEPTABLE ACCEPTABLE
DIREERA					267- 358	ACCEPTAULE
	2	50.1	51.3	39.4- 51.9	42.3- 59.1	ACCEPTABLE
BERYLLIUM	1 2				693- 384 17.4- 24.4	ACCEPTABLE ACCEPTABLE
CADMIUM	1 2	5.10 71.0	5.35 72.0	4.20- 8.85 60.4- 82.5	4.78- 8.27 63.2- 79.7	ACCEPTABLE ACCEPTABLE
COBALT	1 2	435 · 27.3		382- 509 22.6- 32.2	398- 493 23.8- 30.9	
		27.3	21.2	22.5- 32.2	23.6- 30.9	ACCEPIANES
CHRONIUM	1 2					ACCEPTABLE ACCEPTABLE
COPPER	1 2	701 26.2	720 25•2		657- 770 21.3- 29.6	
	2	20.2	23.2	19.9- 31.0	21.3- 29.6	ACCEPTABLE
IRON	1 2	37.6 1210	32.5 1230	22.5- 48.9 1070- 1390	25.8- 45.6 1110- 1350	ACCEPTABLE ACCEPTABLE
MERCURI .	1 2	4.76 37.2	5.01 44.0		4.55- 6.25 35.7- 50.9	ACCEPTABLE ACCEPTABLE
MANGANESE	1 2	551 18.6	551 19.3		438- 609 16.4- 22.6	ACCEPTABLE
			_	17.4- 23.3	100- 2200	
NICKEL	1 2	932 41.4	940 41.3		849- 1020 35.8- 47.5	ACCEPTABLE ACCEPTABLE
LEAD	1 2	31.8 1290	32.2 1344	24.7- 40.7	26.7- 39.7 1190- 1500	ACCEPTABLE ACCEPTABLE

[#] BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN RECESSARY.

PERFORMANCE SYALUATION REPORT

DATE: 12/21/90

WATER POLLUTION STUDY NUMBER MP025

LABORATORY: PA003

ANALYTES					WARRING LIMITS	
TRACE METAL	S IN HICH	CRARSOO	PER LIT	23:		
SELENION	1	27.a	30.0	20.1- 36.3	22.2- 34.7	ACCEPTABLE
	2	127	130	90.7- 155	98.9- 147	ACCEPTABLE
MANADIUM		57.1	53.1	16.6- 69.8	49.7- 56.7	ACCEPTABLE
•	2	246	255	218- 291	228- 281	ACCEPTABLE
ZIXC	1	22.2	25.4	17.6- 36.9	20.1- 34.5	ACCEPTABLE
	2	765		671- 953		ACCEPTABLE
ANTIKONY	3	22.8	24.0	14.4- 31.6	16.6- 29.4	ACCEPTABLE
	4			110- 201		ACCEPTABLE
SILVER	3	1.20	1.20	0.748- 1.61	0.855- 1.50	ACCEPTABLE
	ц	11.5		9.58- 15.2		ACCEPTABLE
THALLIUM	3	10.1	11.0	5.79- 15.7	7.96- 14.7	ACCEPTABLE
	4.	64.5		51.1- 85.1		ACCEPTABLE
ROLYBDENUM	3	9.53	9.58	4.74- 14.3	5.09- 12.9	ACCEPTABL!
	4	59.3	56.0	33.9- 74.7	39.6- 68.9	ACCEPTABL
STRONTIUM	3	. 16.5	15.9	13.5- 21.2	14.5- 29.2	ACCEPTABLE
	4				39.0- 54.7	
TITANIUH	3	259	250	216- 297	227- 286	ACCEPTAUL
	Ħ	50.0	50.9	39.5- 62.1	41.7- 58.9	ACCEPTABL
MINERALS I	S HILLIGR	ANS PER	LITER:	(EXCEPT AS %	OTED)	
PH-UNITS	3	4.64	4.50	4.52- 4.53	4.54- 4.66	ACCEPTABLE
					8.03- 6.44	ACCEPTABL
SPEC. COND.	1	65.5	67.9	57.5- 76.4	59.8- 74.0	ACCEPTABL
(DMHOS/CM AT 25 C)	1 2	752	_			ACCEPTABL

BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCY VALUE WHEN NECESSARY. .

PERFORMANCE EVALUATION REPORT DATE: 12/21/30

Beach Mills

WATER POLLUTION STUDY NUMBER 4P025

ANALYTES	SJAKKZ RIRKUN	XEPOHT BULKY	PRUE VILUE	ACCEPT LIMI				PERFORMANCE EVALUATION
MINERALS I	n wirright	ans per	LITER:	(EXCEPT	K 24 7	OTED)		
TDS AT 180 C	1			11.0-	58.8	16.9-	52.3	ACCEPTA BL
	2	431	412	234-	542	325-	511	ACCEPTABLE
TOTAL HANDNESS	1	11.9	12.0	8.28-	15.6	9.19-	14.6	ACCEPTABLE
(AS CACO3)	2					187-		ACCEPTABL
CALCIUM	1	3.25	3.00	2.49-	3.59	2.63-	3.44	ACCEPTABL
	2	54.5	55.4	43.1-	62.1	49.8-	60.3	ACCEPTABL
AGNESIUM .	1	1.08	1.10	0.329-	1.31	0.976-	1.26	ACCEPTABL
•	2	14.8	15.0	12.9-	17.1	13.4-	16.6	ACCEPTABL
RUIGOS	1	5.44	5.45	4.47-	6.50	4.74-	6.33	ACCEPTABL
	2	50.6	50.7	44.7-	56.0	46.1-	54.5	ACCEPTABL
RUIZZATOS	1	3.04	3.00	2.41-	3.44	2.54-	3.31	ACCEPTABL
	2	25.9	25.0	21.6-	30.9	22.8-	29.8	ACCEPTABL
	1	5.67	6.59	2.62-	9.74	3.51-	8.85	A CCEPTA BL
(AS CACO3)	2	45.4	47.3	39.5-	50.4	-8.C#	49.1	ACCEPTABL
CHLORIDE	1	9.73	8.56	7.19-	11.7	7.75-	11.1	ACCEPTABL
	2	148	142	131-	154	134-	151	ACCEPTABL
FLUORI DE	1							ACCEPTABL
	2	.782	0.)10	0.772-	1.03	0.804-0	. 997	CHECK FOR ERR
SULFATE	1	7.50	A00	5.00-	10.5	5.70-	9.84	ACCEPTABL
	2	84.3	30.0	74.7-	102	73.1-	98.6	ACC EP TABL

SASED UPON THEGRETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PAGE 3

1 8.84 8.76 5.97-10.4 7.38-10.0 ACCEPTABLE 2 1.64 1.60 1.20-1.39 1.29-1.90 ACCEPTABLE

PERFORMANCE EVALUATION REPORT

DATE: 12/21/99

WATER POLLUTION STUDY NUMBER 4P025

ANALYTES			3081	ACCEPTANCE	VARNING	PERFORMANCE
	NUMBER	* X L U E	445050	LISITS		KOLTKUJKYK
NUTRIENTS I	N MILLIGR	ras sur	LITER:			
HITRATE-NITROGEN		3.27	3.20	2.52- 3.85	2.58- 3.69	ACCEPTABLE
	2	. 648	0.550	0.473-0.826	0.515-0.786	ACCEPTABLE
OXTHOPHOSPHATE	1	.191	0.190	0.145-0.235	0.155-0.224	ACCEPTABL
	2	4.73	5.30	4.48- 6.10	4.67- 5.90	ACCEPTABLE
KJELDAHL-NITROGEN	3 (0.20	7.00	5.01- 3.72	5.45- 8.28	NOT ACCEPTABL
	4	33.1	25.7	19.5- 32.1	21.0- 30.6	NOT ACCEPTABL
TOTAL PHOSPHORUS	3 (0.05	9.20	6.32- 9.62	6.72- 9.23	NOT ACCEPTABL
	4	.611	0.525	0.449-0.772	0.488-0.733	ACCEPTABL
DEMANDS IN	MILLIGHAM	s per l	.ITER:			•
COD	. 1	115	121	95.0- 138	101- 133	ACCEPTABL
	2	13.1	13.2	3.16- 28.3	10.7- 25.8	ACCEPTABL
TOC	1	46.9	44.0	10.3- 55.4	42.7- 53.5	A CCEPTA BL
•		6.32	7.20	5.72- 8.96	6.14- 8.54	ACCEPTABL
5-DAY BOD	1	32.8	76.5	45.1- 108	52.9- 100	ACCEPTABL
•	2					NOT ACCEPTABL
SCB.2 IN H	CROGRAMS	PER LIT	TER:			•
PCB-ARUCLOR 1016/1	242 2	5.69	6.50	2.29- 8.35	3.13- 8.01	ACCEPTABL

[#] BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PAGE 4

DATE: 12/21/20

TROGER HOLLAULKYE EDRAROGRE

WATER POLLUTION STUDY NUMBER WP025

LABORATORY: PAGG9 SAMPLE REPORT TRUE ACCEPTANCE NUMBER VALUE VALUE LIMITS RARNING PERFORMANCE EVALUATION ANALYTES LIMITS PCB'S IN OIL IN MILLIGRAMS PER KILOGRAM: PC3 IN OIL- 1254 1 27.7 25.3 4.04-46.7 9.50-41.2 ACCEPTABLE 50.0 1.58- 02.7 12.0- 72.3 PCB IN OIL- 1260 19.3 ACCEPTABLE PESTICIDES IN MICROGRAMS PER LITER: 1.38 CHLORDANE 1.50 0.744- 1.98 0.902- 1.82 ACCEPTABLE 6.56 5.73 3.36- 6.78 4.06- 6.09 ACCEPTABLE ALDRIN 1 -115 0.158 .0409-0.224 .0643-0.201 ACCEPTABLE ·410 0.483 .0955-0.654 0.166-0.583 2 ACCEPIABLE DIELDRIN .122 0.142 .0479-0.218 .0694-0.195 ACCEPTABLE .498 0.508 0.211-0.716 0.275-0.652 ACCEPTABLE DDD .172 0.131 .0535-0.311 .0907-0.279 ACCEPTABLE . 809 0.773 0.406- 1.08 0.491-0.991 ACCEPTABLE DDE . 199 0.217 .0302-0.308 0.118-0.289 ACCEPTABLE .433 0.425 0.173-0.602 0.228-0.547 ACCEPTABLE CUT .143 0.173 .0421-0.306 .0756-0.273 ACCEPTABLE .546 0.553 0.252-0.812 0.323-0.740 ACCEPTABLE HEPTACHLOR .159 0.193 .0630-0.255 .0917-0.231 ACCEPTABLE . 612 0.623 0.125-0.320 0.226-0.819 ACCEPTABLE VOLATILE HALOCARBORS IN MICROGRAMS PER LITER: 1,2 DICHLOROETHANE 14.3 13.3 1 3.79- 13.3 10.0- 17.1 ACCEPTABLE 29.1 26.7 18.3- 36.0 20.5- 33.7 ACCEPTABLE

BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PERFORMANCE EVALUATION REPORT

DATE: 12/21/90

WATER POLLUTION STUDY NUMBER W2025

ANALYTES	REENUR	VALUE	AYFAE⇔	ACCEPTANCE LIMITS	LIBITS	SOKAÇEORKSY KOLTAULAYS
YOLATILE HAL	CCAR BONS	13 510	ROGRAMS	PER LITER:		·
CHLOROFORA		9.76	9.7:1	6.16- 13.4	7.08- 12.5	ACCEPTABLE
	2	38.6	37.4	24.4- 49.7	27.6- 46.4	ACCEPTABLE
1,1,1 TRICHLOROETHAN	E 1	8.19	7.36	4.44- 11.7	5.37- 10.7	ACCEPTABL
	2	59.7	53.3	35.9- 77.2	41.1- 72.0	ACCEPTABLE
TRICHLOROETHENE		10.5	10.5	5.43- 13.7	7.36- 12.8	ACCEPTABL
	2	53.5	47.4	30.1- 50.5	34.0- 55.9	ACCEPTABL
CARBONTETRACHLORIDE	1 '	7.08	5.31	3.72- 9.38	4.52- 9.18	ACCEPTABL
	2	59.5	54.7		39.6- 71.6	ACCEPTABL
TETHACHLORORTHERE	1	12.3	11.7	5.64- 16.4	7.88- 15.2	ACCEPTABL
	. 2	53.8	54.0	32.1- 74.1	37.4- 68.8	ACCEPTABL
Y KAHTSM OSOJHOI GCMGHE	ı	10.2	10.1	6.59- 13.3	7.51- 12.3	ACCEPTABL
	2	68.0	52.5	43.3- 83.7	46.5- 79.5	ACCEPTABL
DIBHOMOCHLOROMETHANE	1	13.2	12.5	7.53- 17.8	8.93- 16.5	ACCEPTABL
	2	47.9	44.7	27.1- 61.1	33.2- 57.0	ACCEPTABL
BROMOFORM	1	15.5	14.4	7.73- 19.5	9.26- 18.0	ACCEPTABL
	2	70.2	55.1	40.4- 95.6	47.5- 88.5	ACCEPTABL
HETHYLENE CHLORIDE	1	13.5	12.3	6.27- 17.4	7.70- 16.0	1671307
•			42.5			ACCEPTABL
CHLOROBENZENE			13.9	8.37- 18.7	10.2- 17.4	ACCEPTABL
			63.0	43.1- 79.7	47.8- 75.0	ACCEPTABL

[#] HASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY...

BENZENE

1 17.9 17.8 11.9-23.7 13.4-22.2 ACCEPTABLE 2 81.7 32.0 55.7-107 62.2-100 ACCEPTABLE

PERFORMANCE EVALUATION REPORT

DATE: 12/21/90

FATER POLLUTION STUDY NUMBER #2025

LAUGRATORY: PAGGS

(IN MS/L)

	SAMPLE	REPORT	TRUE	RCCEPTANCE	WARNING	PERFORMANCE

SETYLKK RUMBER VALUE VALUE≎ LIMITS LIMITS EVALUATION VOLATILE ARCHATICS IN MICROGRAMS PER LITER: ETHYLBENZERR 9.19 7.60 5.43- 12.9 5.38- 12.0 ACCEPTABLE 39.1- 76.8 43.9- 71.3 59.1 53.4 ACCEPTABLE 9.93- 15.8 TOLUENE 12.5 12.3 3.30- 16.3 ACCEPTABLE 69.8- 112 90.7 62.5- 119 ACCEPTABLE 93.3 1,2-DICHLOROBERZENE 12.4 13.5 8.69- 17.9 9.90- 16.7 ACCEPTABLE 39.1- 77.2 44.1- 72.2 2 53.6 57.2 ACCEPTABLE 1, 3-DICHLOROBENZENE 19.0 13.7 12.1- 26.0 13.9- 24.2 ACCEPTABLE 34.0- 67.5 2 51.1 52.5 38.4- 63.1 ACCEPTABLE 1,4-DICHLOROBENZENE 14.1 15.3 9.55- 21.1 11.1- 19.6 ACCEPTABLE 45.9 43.4 31.8- 65.7 36.2- 61.2 ACCEPTABLE MISCELLANEOUS PARAMETERS: .503 TOTAL CYANIDE 0.540 0.392-0.631 0.428-0.645 ACCEPTABLE 1 .079 (IN AS/L) 2 0.110 .0613-0.146 .0720-0.135 ACCEPTABLE 1, 17.2 NON-FILTERABLE RESIDUE 15.5 11.9- 21.2 13.1- 20.0 ACCEPTABLE 34.9- 50.0 36.7- 48.1 ACCEPTABLE (IN MG/L) 2 42.6 42.4 SEASE DIL AND GREASE 38.6 39.0 23.2- 45.1 26.6- 42.5 ACCEPTABLE 1 (IN MG/L) 18.3 20.0. 10.6- 25.4 - 12.4- 23.6 · ACCEPTABLE 2 TOTAL PHENOLICS ACCEPTABLE 1 2.64 1.53- 4.56 2.01- 4.27 3.14 (IN MG/L) .322 ACCEPTABLE 0.149-0.596 0.205-0.539 0.372 IOTAL RESIDUAL CHLORINE 1 .155 0.175 D.L.-0.351 .0378-0.304 ACCEPTABLE

1.65

2

1.47

PAGE 7 (LAST PAGE)

1.07- 1.99

1.19- 1.87

ACCEPTABLE

BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY. D.L. STANDS FOR DETECTION LIMIT

REGION 3 ORGANIC PERFORMANCE EVALUATION SAMPLE INDIVIDUAL LABORATORY SUMMARY REPORT FOR OB 3 FY 91

4.4.4.4

LABORATORY: Lancaster Laboratories (PA)

PERFORMANCE: ACCEPTABLE - No Response Required
RANK: Above = 6 Same = 0 Below = 19

% SCORE: 95.6 REPORT DATE: 06/10/97 MATRIX: WATER

										DATES	: WATER
_		CONFIDENCE INTERVALS LABORATOR									
		UAR	NING			LABOR			PROGRAM	DATA '	
	COMPOUND	LOWER	UPPER	LOWER	ION UPPER	CONC		#LABS	#LABS	#LABS	TOTAL
	TCL VOLATILE .				OFFER	1 COME	9	MIS-QNT	NOT-ID	ID-CPD	#LABS
	CHLOROMETHANE	30	93	20	. 100	50		•	_		
	1,1-DICHLOROETHANE	10	12	10	12	10		2	0	35	35
~	CHLOROFORM	62	78	60	80	78		2	0	35	35
	2-BUTANONE	43	120	32	130	94		3	0	35	35
	CIS-1,3-DICHLOROPROPENE	60	97	54	100			0	1	34	35
	BROMOFORM	57	76	54	78	78		3	1	34	35
_	2-HEXANONE	91	250	67	270	64		5	0	35	35
	1,1,2,2-TETRACHLOROETHANE	120	160	110	160	180		5 .	0	35	35
	CHLOROBENZENE	34	. 43	33	45	140		5	0	35	35
	STYRENE	160	200	150	210	42		2	0	35	35
	XYLENES (TOTAL)	74	93	71		180	_	3	0	35	35
_		, ,	, ,		100	95	\$	5	2	33	35
	TCL SEMIVOLATILE										
	BIS(2-CHLOROETHYL)ETHER	14	22	13							
	2-CHLOROPHENOL	19	29	18	23	19		5	1	34	35
	1,3-DICHLOROBENZENE	20	34	18	30	25		7	0	35	35
	NITROBENZENE	34	59		43	27		0	0	35	35
	2-HITROPHENOL	24	40	31 22	63	49		3	1	34	35
	2,4-DIMETHYLPHENOL	38	66		43	38		7	0	35	35
_	2,4,5-TRICHLOROPHENOL	62	94	34 57	70	37	\$	6	0	35	35
	DIMETHYL PHTHALATE	11	110		110	73		3	1	34	35
	ACENAPHTHYLENE	25	40	10	120	69		0	6	29	35
	2,4-DINITROPHENOL	50	_	22	42	32		2	0	35	35
·	4,6-DINITRO-2-METHYLPHENOL	NU	140	50	150	130		1	0	35	35
\sim	4-BROMOPHENYL PHENYL ETHER	22	NU	NU	NU	36		0	2	33	35
	PHENANTHRENE .	11	41	20	44	31		1	0	35	35
	PYRENE	74	18	10	20	16		0	0	35	35
	BEHZO(K)FLUORANTHENE	NU.	120	66	150	66	\$	0	0	35	35
	INDENO(1,2,3-CD)PYRENE	10	NU	NU	WU	17		0	10	25	35
		10	18	10	23	17		1	2	33	35
	TCL PESTICIDES										
~	DELTA-BHC .	0.082	0.18	0.067	0.19			_			
	HEPTACHLOR	0.091	0.16	0.081	0.17	0.13		3	0	35	35
	ALDRIN	0.26	0.42	0.23		0.11		5	0	35	35
	HEPTACHLOR EPOXIDE	0.27	0.38	0.25	0.44	0.26		2	0	35	35
	4,41-DDE	0.31	0.42		0.4	0.3	_	6	3	32	35
_	ENDOSULFAN SULFATE	0.38	0.72	0.29	0.44	0.3	\$	4	- 0	35	35
	AROCLOR-1260	1.3	2.3	0.33 1.1	0.77 2.4	0.52 1.5		4 5	1 4	34 31	35
	NON-TCL VOLATILE							•	•	31	35
~	50.10										
	EPICHLOROHYDRIN					0			35	•	30
	PROPANE, 1, 2-DIBROMO-2-CHLORO-					ō			33 16	10	35
	TOLUENE, 2-CHLORO-					ŏ	2		4	19 31	35 35
	HON-TCL SEMIVOLATILE										
	BENZOIC ACID					100			_		-
						100			7	28	35

REGION 3 ORGANIC PERFORMANCE EVALUATION SAMPLE INDIVIDUAL LABORATORY SUMMARY REPORT FOR OB 3 FY 91 .

LABORATORY: Lancaster Laboratories (PA)

PERFORMANCE: ACCEPTABLE - No Response Required
RANK: Above = 6 Same = 0 Below = 19

X SCORE: 95.6 REPORT DATE: 06/10/91

MATRIX: WATER

		CONFIDEN			΄, ι	ABORAT			PROGRAM	DATA	
OHPOUND	LOVER	UPPER	ACT LOWER	UPPER	0	DATA OHC	•	#LABS THD-21H	2BAJ# DI-TOH	TLABS	TOTAL #LABS
DIBENZOTHIOPHENE	•					12			1	34	35
'CL VOLATILE (Contaminants)											
TRICHLOROETHENE						6 1			25 17	10 18	35 35
CL SEMIVOLATILE (Contaminants)											
DIETHYLPHTHALATE DI-N-BUTYLPHTHALATE IS(2-ETHYLHEXYL)PHTHALATE						1 2 1			34 32 30	1 3 5	35 35 35
MON-TCL VOLATILE (Contaminants)					•						
HKHOLM -PROPANOL			•			15 140	С		29 16	6 19	35 35
NON-TCL SEMIVOLATILE (Contaminants)											
AKNONA NIKNONA NIKNONA NIKNONA NIKNONA			٠			2 2 2 6			25 30 32 33 34	10 5 3 2	35 35 35 35 35 35
NKNOWN WKNOWN WKNOWN CARBOXYLIC ACID						4 3 3			34 34 34	1	35
"MKHOUM CARBOXYLIC ACID ESTE HKHOUM COMPOUND, AROMATIC HKHOUM PHTHALATE			•			37 4 12			34 34 34	; 1 1	35 35 35 35

OF TCL COMPOUNDS NOT-IDENTIFIED: 0 TOF TEL COMPOUNDS HIS-QUANTIFIED: 0 # OF TCL CONTAMINANTS: 0

OF NON-TCL COMPOUNDS NOT-IDENTIFIED: 1
_OF NON-TCL CONTAMINANTS: 1

Program Summary Data (cont.):

Header

Definition

LABS NOT-ID:

The number of CLP contractors who did not identify a TCL or non-TCL compound added to the PEM.

LABS ID-CPD:

The number of CLP contractors who identified a TCL or non-TCL compound in the PEM.

TOTAL # LABS:

The number of CLP contractors who analyzed the PEM.

ILSR CODES:

The following codes are used on the ILSR.

- U -- Compound analyzed for but not detected.
- & -- Compound not identified -- points deducted for identification.
- X -- Compound correctly identified but the reported value is not within the action limit -- points deducted for quantification.
- \$ -- The reported value for the compound is not within the warning limit but is within the action limit -- points not deducted.
- C -- Contaminant -- points deducted.
- CO -- Contaminant which may have been introduced during preparation of the PEM or during shipment -- points not deducted.
- NS -- Data required but not submitted -- points deducted.
- NR -- Data not required.
- NU -- Data not used; insufficient amount of usable data for scoring submitted by the contractors.

DEPARTMENT OF HEALTH & HUMAN SERVICES



Centers for Disease Control National Institute for Occupational Safety & Health Robert A. Taft Laboratories 4676 Columbia Parkway Cincinnati OH 45226-1998

M. LOUISE HESS LANCASTER LABORATORIES, INC. 2425 NEW HOLLAND PIKE LANCASTER PA 17601 August 26, 1991

Dear M. LOUISE HESS:

Enclosed are your results from the Proficiency Analytical Testing (PAT) Program for Round 106. The following table shows the Relative Standard Deviations for reference laboratories:

ROUND 106 RELATIVE STANDARD DEVIATIONS

Contaminant	Number of Ref. Labs	Sample 1	Sample 2	Sample 3	Sample 4
CADMIUM (CAD)	73	04.23%	04.50%	04.76%	04.04%
CHROMIUM (CHR)	73	07.78%	07.15%	08.33%	07.76%
LEAD (LEA)	73	03.34%	03.67%	03.47%	03.05%
SILICA (SIL)	71	27.36%	21.27%	18.67%	22.25%
ASBESTOS (ASB)	73	19.55%	17.26%	18.24%	16.56%
BENZENE (BNZ)	73	06.46%	04.79%	04.07%	04.64%
O-XYLENE (OXY)	73	05.10%	04.89%	04.77%	04.59%
TOLUENE (TOL)	73	05.79%	04.05%	04.30%	03.68%

The Relative Standard Deviations are calculated from non-transformed Winsorized reference laboratory data.

PAT Round 107 is scheduled for shipment on October 1, 1991, and the organic solvents this round will be carbon tetrachloride (CTC), 1,2-dichloroethane (DCE), and trichloroethylene (TCE). Metals this round will include cadmium (CAD), lead (LEA) and zinc (ZIN). Also, silica will have a mix of talc and coal mine dust as the background, and the asbestos will be chrysotile this time.

Sincerely yours,
Jensen H. Groff
Research Chemist
Quality Assurance and Statistics
Activity
Division of Physical Sciences and
Engineering

Enclosure

17601001 M X A O
M. LOUISE HESS
LANCASTER LABORATORIES, INC.
2425 NEW HOLLAND PIKE
LANCASTER. PA 17601

PROFICIENCY ANALYTICAL TESTING PROGRAM LABORATORY PERFORMANCE - ROUND 106

LANCASTER, P	A 17601	!		RND106 STATIS	STICAL	SUMMAR	łΥ		LAI	BORATOR	/ RESU	LTS	
	CONTAMINANT	SAMPLE	REF.	PERFORMANCE LIMITS	NO. LABS	HI	ITLIERS LO	TOT	R106 DATA	PROF RATING			UMMARY 105 106
METALS	CADMIUM (CAD)		0.0122	0.0108-0.0138	383	,-		22	0.0120				
WEINES	(MG)	2	0.0123	0.0087-0.0138	363	11 15	11 11		0.0020	-			
	(MG)	3 1	0.0061			10	15	25					
	İ	4		0.0147-0.0186		6	20	26					
	CHROMIUM (CHR)		0.1185	0.0909-0.1461	379	7	8	15	0.1158				
	(MG)	2	0.0601			4	13	17	0.0573	1			
		3	0.1979	0.1485-0.2474		5	13	18	0.2000	j			
		4	0.1494	0.1146-0.1842		7	17	24	0.1525	ĺ			
	LEAD (LEA)	1		0.0541-0.0660	387	13	28		0.0598				
	(MG)	2	0.0300			21	22		0.0300				
		3		0.0761-0.0937		6	25		0.0855				
	}	4	0.0494	0.0449-0.0538		21	28	49	0.0495				
SILICA	SILICA (SIL)	1		0.0304-0.1789	107	1	2	3		-		_	
	(MG)	2 !		0.0424-0.1684		0	4	4	ł	!	-	-	
	1	3		0.0675-0.2106		0	10	10	l	!	-	_	<u> </u>
	i .	4	0.1353	0.0643-0.2851		0	5	5	l				
ASBESTOS	ASBESTOS (ASB)	1	231.1	115.8- 385.9	1302	58	11	69		P			
	(F/MM2)	2	408.5	224.1- 647.8		61	19	80		1			
	<u> </u>	3 4	805.6 657.6	422.2-1311.8 368.8-1029.2		54 40	12 20	66 60		1			
0000000	051135115 (51.7)								0.0013				
ORGANIC SOLVENTS	BENZENE (BNZ)	1		0.0746-0.1105	376	16	10		0.0917	P [
20FAEM12	(MG)	2		0.1519-0.2028 0.1988-0.2541		13	15 14		0.1710	ł			
	}	3 4		0.1988-0.2541		20 14	10	24		ŀ			
								1		i			
	O-XYLENE (OXY)	1	1.6014		374	10	18		1.6149	!			
	(MG)	2	1.2698	1.0834-1.4562		. 9	18		1.2261	1			
	!	3	1.0270			12	12		1.0094	ļ			
		4	0.7036	0.6068-0.8005		15	17	32	0.6967				
	TOLUENE (TOL)	1	0.7084	0.5854-0.8315	376	8	7	15	0.7138	i			
	(MG)	2		0.8752-1.1171		18	17		0.9733	į			
	1 ' '	3		1.0568-1.3702		14	17		1.2054	Ì			
	İ	4 1		1,1471-1,4322		21	23		1.2997	i			

OVERALL:

1452

P

PROFICIENCY: P = PROFICIENT

RATINGS NP =# OF TIMES N

NP =# OF TIMES NON-PROFICIENT
- =ANALYSIS NOT PERFORMED

OR NOT RATED

OUTLIER: BLANK =ANALYSIS ACCEPTABLE SUMMARY - =ANALYSIS NOT PERFORMED

HI =HIGH OUTLIER

LO =LOW OUTLIER

NOTE: ONLY ONE PROFICIENCY RATING IS GIVEN FOR METALS AND ONLY ONE PROFICIENCY RATING IS GIVEN FOR ORGANIC SOLVENTS.

Section No. 12 Page 14 of 15

15:38 TUESDAY, AUGUST 20, 1991 1110

PROFICIENCY ANALYTICAL TESTING PROGRAM ROUNDS 103 - 106 STANDARD DEVIATION PLOTS

17601001 M X A O
M. LOUISE HESS
LANCASTER LABORATORIES, INC.
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601

		METALI	METAL2	METAL3	SILICA	ASBESTOS	SOLVENTI	SOLVENT2	SOLVENT3
ROUND	SAMPLE	-SD +SD 54321012345	-SD +SD 54321012345	-SD +SD 54321012345	-SD +SD 54321012345	-SD +SD 54321012345	-SD +SD 54321012345	-SD +SD 54321012345	-SD +SD 54321012345
103	1	·	•	•		•	•	•	•
	2	;		:	:	:	! •:	:	:
	3		•	i i	:	;		!	
		:	:	:		i	:	:	:
	4		•		:	•			
104	1	•	•	•		•			
	2	÷	:		:	:	.	;	
	3	;	•	;	:	:		•	:
	4	• :		÷		•	• :		:•
	·				·	•	•		•
105	1	٠.		*	•			•	*
	2	÷	i	i	:	•	•	•	•
	3	•:	÷	÷		•	•	•	i
	4	*.	÷	÷		* .	*:	;	•:
		•	•	·	·	•	•	•	•
106	1	•	•	•		•	•	•	•
	2	÷	÷	•		. •	*	•	•
	3	÷	÷	÷		.•	÷	i	÷
	4	÷	÷	÷		÷	÷	÷	÷
		·	·		·	•	•		•
S USED	- R103: R104:	CAD CAD	CHR LEA	LEA ZIN	SOLVENTS	USED - R103: R104:	CTC MCM	DCE PCE	TCE TCE
	R105: R106:	CAD CAD	LEA CHR	ZIN Lea		R105: R106:	CFM BNZ	CTC OXY	DCE TOL

Section No. 12 Page 15 of 15

Section No. 13 Revision No. Date: 11/14/91 Page 1 of 2

13. Preventive Maintenance

In order to ensure timely production of data, Lancaster Laboratories, Inc. (LLI) schedules routine preventive maintenance of instruments based on manufacturer's recommendations. Maintenance of the laboratory instruments is the responsibility of the technical group using the equipment in conjunction with our in-house equipment maintenance group. A schedule of routinely performed instrument maintenance tasks is attached as Table 13-1. All preventive maintenance, as well as maintenance performed as corrective action, is recorded in instrument logs.

Critical spare parts are kept in supply at the laboratory by the equipment maintenance group. Most items not kept in stock at the laboratory are available through overnight delivery from the manufacturer. In addition, LLI maintains multiple numbers of most of the critical instruments used in our laboratory operations. A recent equipment inventory may be found in the Qualification Manual. Because we are a large laboratory with redundant capacity, the problems of instrument downtime are minimized.

Section No. 13 Revision No. Date: 11/14/91 Page 2 of 2

Table 13-1 Preventive Maintenance Schedule

Instrument	Preventive Maintenance	Frequency
GC	Injection Syringe Septum change Column maintenance Clean detector	Weekly Weekly AN AN
HPLC	Reproducability Check Lubricate moving parts Inspect & clean line filter Clean Check valve & pump head Replace plunger seal Replace syringe plunger tip Check lamp intensity	6 months 3 months 6 months 6 months 6 months 6 months 6 months

^{*} AN means as needed. Any of these items may be performed more frequently if response during operation indicates this is necessary.

Section No. 14
Revision No.
Date: 11/14/91
Page 1 of 3

14. Specific Routine Procedures Used to Assess Data Precision, Accuracy and Completeness

<u>Precision</u> - Precision refers to the reproducibility of a method when it is repeated on a second aliquot of the same sample. The degree of agreement is expressed as the Relative Percent Difference (RPD). The RPD will be calculated according to the following equation:

$$RPD = \frac{D_2 - D_1}{(D_1 + D_2)/2} \times 100$$

D₁ = First sample value
D₂ = Second sample value (Duplicate)

Duplicates will be run on at least 5% of the samples. Acceptance criteria shall be based on statistical evaluation of past lab data. (See Section No. 11.) All Quality Control sample results are entered into the computer and compared with acceptance limits. In addition, there is a monthly review of values on the computer QC system. Data obtained from quality control samples is entered onto our computer system which charts the data, and calculates a mean and standard deviation on a monthly basis. The Quality Assurance Department then reviews this data for trends which may indicate analytical problems. The control charts are graphical methods for monitoring precision and bias over time.

Accuracy - Accuracy refers to the agreement between the amount of a compound measured by the test method and the amount actually present. Accuracy is usually expressed as a percent Recovery (R). Recoveries will be calculated according to the following equations:

Section No. 14 Revision No. Date: 11/14/91 Page 2 of 3

Surrogate Recovery = $\frac{Qd}{Qa}$ x 100

Qd = quantity determined by analysis Qa = quantity added to sample

Matrix Spike Recovery = $\frac{SSR - SR}{SA} \times 100$

SSR = Spiked Sample Results

SR = Sample Results

SA = Spike added

Laboratory Control Sample Recovery = $\frac{LCS Found}{LCS True} \times 100$

Surrogate standards are added to each sample analyzed for Spikes and Laboratory Control Samples will be run on at least 5% of the samples (each batch or SDG, < 20 samples). Acceptance criteria for the accuracy recoveries shall be based on statistical evaluation of past lab (See Section No. 11.) The computer is programmed to compare the individual values with the acceptance limits and inform the analyst if the results meet specification. If the results are not within the acceptance criteria, corrective action suitable to the situation will be taken. This may include, but is not limited to, checking calculations and instrument performance, reanalysis of the associated samples, examining other QC analyzed with the same batch of samples, and qualifying results with documentation of any QC problems in the Case Narrative.

Where available, EPA Quality Control materials are run at least quarterly to ensure accuracy of the analytical procedure. Repetitive analysis of a reference material will also yield precision data. Accuracy information determined from reference materials is valuable because variables specific to sample matrix are eliminated.

Section No. 14 Revision No. Date: 11/14/91 Page 3 of 3

The QC program is capable of charting data for surrogates, spikes, control materials and reference materials. The Quality Assurance Department reviews these charts for any indication of possible problems (ie shift in the mean and standard deviation).

Completeness - Completeness is the percentage of valid data acquired from a measurement system compared to the amount of valid measurements that were planned to be collected. The objective is analysis of all samples submitted intact, and to ensure that sufficient sample weight/volume is available should the initial analysis not meet acceptance criteria. The laboratory's Sample Management System will assign a unique identification number to the sample which tracks and controls movement of samples from the time of receipt until disposal. generated will be recorded referencing the corresponding sample identification number. The completeness of an analysis can be documented by including in the data deliverables sufficient information to allow the data user to assess the quality of the results. This information will include, but is not limited to, summaries of QC data and sample results, chromatograms, spectra, and instrument tune and calibration data. Additional information will be stored in the laboratory's archives, both hard copy and magnetic tape.

Section No. 15 Revision No. Date: 11/14/91 Page 1 of 3

15. Corrective Action

Whenever any of the data generated falls outside of the established acceptance criteria outlined for instrument tune and calibration (Section No. 8) and Internal QC (Section No. 11), the cause of this irregularity must be investigated, corrected and documented. The documentation will be used to prevent a recurrence of the problem and to inform management of the situation.

If the results are not within acceptance criteria, the appropriate corrective action will be initiated. This may include, but is not limited to, checking calculation and instrument performance, reanalysis of the associated samples, examining other QC analyzed with the same batch of samples, and qualifying results with a comment stating the observed deviation.

A Standard Operating Procedure is in place which outlines the procedures to be followed when quality control data for an analysis falls outside of previously established acceptance limits. All QC data must be entered onto the computerized QC system promptly after its generation and daily "out-of-spec" data is reported via this system. Any data outside the acceptance criteria will be reviewed by the Quality Assurance Department. Where appropriate, the Quality Assurance Department will place outliers in one of three categories:

A. Marginal Outlier

Data that are outside the 95% confidence interval but within the 99% confidence interval. This category may also be used for QC samples subject to matrix interferences or sample inhomogeneity.

Section No. 15 Revision No. Date: 11/14/91 Page 2 of 3

B. Outlier

Data outside the 99% confidence interval and/or observable trends such as a shift in mean and standard deviation.

C. Extreme Outlier

Such data would indicate the system is out of control and no results should be reported to clients; an example would be more than one reference or control falling outside the 99% confidence interval.

The daily out-of-spec reports are then distributed to Group Leaders or their QC Coordinator who will check all supporting data and document their findings and any corrective action taken. Documentation of QC Data will be filed in the departmental QC notebook. In the case of Outliers or Extreme Outliers the Quality Assurance Department may issue a formal request for investigation and corrective action (see sample form that follows). The Quality Assurance Department is responsible for initiating the corrective actions, insuring that the actions are taken in a timely manner, and that the desired results are produced.

The Quality Assurance Department is also responsible for conducting periodic audits which ensure compliance with laboratory SOP's and assist in identifying and correcting any deficiencies. These audits may entail observation as procedures are carried out or a review of records to demonstrate traceability and compliance with all documented record keeping procedures. Follow-up audits verify that proper corrective action has been taken for the identified discrepancy.

Section No. 15 Revision No. Date: 11/14/91 Page 3 of 3

		No
	741	ESTIGATION AND CORRECTIVE ACTION FOR QC OUTLIERS
Part I	(to	be filled out by QA Director)
	1.	Date
	2.	LLI sample number(s) involved
	3.	Nature of QC outlier
	4.	Check if investigation must be complete before reporting further data to clients.
		Signed
		Quality Assurance Director
Part I	Ξ	
	1.	Steps taken to investigate outlier:
	2.	Explanation of probable cause of outlier:
	3.	Steps taken to prevent future occurrence:
	4.	Name of analyst who performed work:
	5.	Signed Date

Section No. 16
Revision No.
Date: 11/14/91
Page 1 of 1

16. Quality Assurance Reports to Management

Reports of quality status from the Quality Assurance Department to management are made frequently and in various forms. All results from internal or external performance evaluation samples are circulated to management. A report of each audit performed is prepared and copied to management. Monthly summaries of data obtained from analysis of quality control check samples are generated via the computerized sample management system. These summaries include mean and standard deviation to aid in assessment of data accuracy and precision. Forms summarizing problems which require investigation and corrective action are completed by Group Leaders and circulated to management. Through these channels, laboratory management is kept apprised of QA/QC activities.

Any problems or unusual observations that occur during the analysis of samples for a specific project will be listed on the laboratory report and/or in the case narrative delivered with the data package. The items often discussed in this manner include samples with surrogate recovery outside of the acceptance criteria and samples with matrix problems requiring dilution and causing increased detection limits. Where applicable, any corrective action attempted or performed to address the problem will also be presented.

The laboratory will contact the client for direction regarding major problems such as samples listed on the chain of custody but missing form the shipping container, samples which arrive broken or are accidentally broken in the laboratory, and samples with severe matrix problems. The client will be contacted if it is necessary to change any item in the original project plan.

Appendix A

Example Reporting Forms

Tier I Data Package

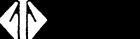
Title Page Table of Contents Sample Analysis Request Form, Field Chain of Custody Internal Chain of Custody Laboratory Chronicle Method Summary/References Analytical Reports for Samples and QC Samples Case Narrative QC Summary GC/MS tuning summary Surrogate recovery summary Blank results Matrix spike/matrix spike duplicate/duplicate results LCS results (if applicable) Internal standard area summary (GC/MS) Sample Data All raw sample data including instrument printouts (i.e., chromatograms, quant. reports, spectra, etc.) Standards Data Initial calibration summary and supporting raw data Continuing calibration summary and supporting raw data Standardization data Raw QC Data

Raw tune data (GC/MS)

Blank raw data

Matrix spike/matrix spike duplicate/duplicate raw data LCS raw data (if applicable)

Extraction/Digestion Logs



ANALYSIS REPORT

2425 New Holland Pike, Lancaster, PA 17601-5994

Lancaster Laboratories, Inc. 2425 New Holland Pike

Lancaster, PA 17601-5994 elitizSamplerNowAOzi/28128zezezez Date Reported 10/28/91 Date Submitted 10/16/91 Discard Date 10/16/91

Primary Air Sample 50 min. 0.2 L/min 10 L 25C

المقاعات البيأ ومخلا كالكرا

P.O. Rel.

	RESULT	LIMIT OF	
ANALYSIS	AS RECEIVED	QUANTITATION	LAB CODE
Acetone	0.005 J mg/tube	0.008	900100000
Chlorobenzene	N.D. mg/tube	0.01	900200000
Chloroform	N.D. mg/tube	0.02	900300000
1,1-Dichloroethane	N.D. mg/tube	0.01	900400000
l,1-Dichloroethylene	N.D. mg/tube	0.01	900500000
Ethyl Benzene	0.016 mg/tube	0.006	900600000
Methyl Ethyl Ketone	N.D. mg/tube	0.009	900700000
Methyl Isobutyl Ketone	0.006 J mg/tube	0.009	900800000
Methylene Chloride	N.D. mg/tube	0.02	900900000
Tetrachloroethylene	N.D. mg/tube	0.04	901000000
Toluene	0.024 mg/tube	0.008	901100000
I,1,1-Trichloroethane	N.D. mg/tube	0.02	901200000
1,1,2-Trichloroethane	N.D. mg/tube	0.02	901300000
Trichloroethylene	N.D. mg/tube	0.02	901400000
Xylenes	0.048 mg/tube	0.009	901500000
Acetone in Air	0.2 J ppm	0.4	901600000
Chlorobenzene in air	N.D. ppm	0.3	901700000
Chloroform in air	N.D. ppm	0.5	901800000
l,l-Dichloroethane in air	N.D. ppm	0.3	901900000
l,l-Dichloroethylene in air	N.D. ppm	0.3	902000000
Ethyl Benzene in air	0.4 ppm	0.1	902100000
Methyl Ethyl Ketone in air	N.D. ppm	0.3	902200000
Methyl Isobutyl Ketone in air	0.15 J ppm	0.2	902300000
Methylene Chloride in air	N.D. ppm	0.6	902400000
Tetrachloroethylene in air	N.D. ppm	0.6	902500000
Toluene in air	0.6 ppm	0.4	902600000
l,l,l-Trichloroethane in air	N.D. ppm	0.4	902700000
1,1,2-Trichloroethane in air	N.D. ppm	0.4	902800000
Trichloroethylene in air	N.D. ppm	0.5	902900000
Xylenes in air	1.1 ppm	0.2	903000000

1 COPY TO Kathy Loewen

The American Association for aboratory Accreditation hemical, Biological & Environmental elds of testing.

Questions? Contact Environmental Client Services at (717) 656-2301 0.00 129 00649 000000

independent Laboratories, Inc.

See Reverse Side For Explanation Of Symbols And Abbreviations And **Our Standard Terms And Conditions** Respectfully Submitted Lancaster Laboratories, Inc. Reviewed and Approved by:

Richard C. Entz, Group Leader Misc. Organics/Ind. Hygiene



ANALYSIS REPORT

Lancaster Laboratories INCORPORATED

2425 New Holland Pike, Lancaster, PA 17601-5994 (717) 656-2301

00649

THE THE PROPERTY OF THE PARTY O

Lancaster Laboratories, Inc. 2425 New Holland Pike Lancaster, PA 17601-5994

Date Reported 10/28/91
Date Submitted 10/16/91
Discard Date 10/16/91

LAB CODE

900100000

900200000

#2 Air Sample
100 min @ 0.1 L/min 10 liters 25 C

P.O. Rel.

ANALYSIS Phenol Phenol in Air

THE PROPERTY OF THE PROPERTY O

.

100

RESULT LIMIT OF
AS RECEIVED QUANTITATION
N.D. mg/tube 0.008
N.D. ppm 0.2

1 COPY TO Kathy Loewen

The American Association for aboratory Accreditation Chemical, Biological & Environmental lets of testing.

ember American Countil of

Questions? Contact Environmental Client Services at (717) 656-2301 129 00649 0.00 000000

See Reverse Side For Explanation Of Symbols And Abbreviations And Our Standard Terms And Conditions Respectfully Submitted Lancaster Laboratories, Inc. Reviewed and Approved by:

Richard C. Entz, Group Leader Misc. Organics/Ind. Hygiene



Method Blank

Selected VOCs on charcoal tubes

*** BLANK INFORMATION ***

Matrix..... Air/Charcoal Tube

Batch Number....:
Injection number...:
Analysis date...:

222222222	=======================================		**********		######################################		
Sample Inf					amination Information		1
LLI	Client	Analy	/sis		1	Blank	L00
Sample No.	Designation	Date	Time	CAS Number	Compound	Result	(mg)
		:======================================		2222222 	======================================	:======== ND	0.008
i	i		i	ii	Chlorobenzene -	ND	0.014
i	i			i i	Chloroform	ND	0.022
i	1			ii	1,1-Dichloroethane	ND	0.013
i	1			ii	1,1-Dichloroethene	ND	0.012
i	i			ii	Ethyl benzene	ND	0.006
i	i		i	ii	Methyl ethyl ketone	DND	0.009
i	i			ii	Methyl isobutyl ketone	ND	0.009
i	i i		i	ii	Methylene chloride	סא	0.022
i	i i		i	ii	Tetrachloroethylene	ND ND	0.042
i	į į			ii	Toluene	DMD	0.008
i	i		j	ii	1,1,1-Trichloroethane	ם א	0.022
İ	İ		i	ii	1,1,2-Trichloroethane	ND	jo.022 j
İ	į :		į	11	Trichloroethylene	ND	0.024
İ	İ		İ	İİ	Xylenes (sum of isomers)	ND	0.009
İ	İ			İ	İ	ļ	į į
1	1		1]]	1	ļ	1 1
ļ	ļ		ļ	!!	1	Į.	! [
Į	į		!	! !	Ì	ļ	[[
1	ļ		ļ		1	1	1 1
ļ	I		1		1	}	1 !
ļ	Ţ		!	!!		ŧ	1 1
1	1			11	1	I	1 1
ļ			ļ]]	1]	1 1
1	ļ	<u> </u>	!	11	1	1	-
ļ.	!		!	!!	<u>I</u>	!	!!
ļ	ļ	[ļ	!!		1	
1	1	1	1	!!]	1	1 1
ļ		[1	!!		ļ	j j
1	ļ]		<u>[</u>]		ļ	1 1
!	!		!	!!		ļ	
!	!	!	!	ii		!	!!!
ļ	į	•	!	!!	ļ	Ĺ	
!	1	!	!	!!	ļ	ļ	!!!
ļ	Į.	!	!	!!		!	[]
· I	1	Į.	1	11	1	1	} }

Comments:

LOQ = Limit of Quantitation

ND = None Detected

NR = Not Reported



Surrogate Recovery

Batch Number: SDG Number: Selected VOCs on Charcoal Tubes

*******		********	*======		
	Client				Comment
	Designation			Butanol #	
1			 		
	1) 	!]	i	i
i	į	İ	i	į	İ
i	i	i	i	İ	j
İ	İ	•	ĺ	•	
1	1	[ļ	
ļ	1]	ļ	!	!
ļ	ļ	1		[! !
-		!	1	}]
i I	! 	i I	1	ł 	(
i	i	i	i	i	į
i	i	i	i	ì	i
i	İ	j	j	j	j
1	I	1	1	1	· .
	Ì	!	ļ	ļ]
	!	ļ	Į.	1	!
ļ	!	!	1	ļ	1
l i	1	1	1	1	(!
1		i	1	ĺ	ŧ.
i	i	i	i	į	i i
j	i	i	i	j	j
İ	İ	İ	ĺ	İ	1
!	Ţ	!	ļ .	ļ	
1	!	ļ	!	ļ	
1	1		!	!	
1	1	1	1	1	1
1		1	-	•	1
i	i	i	i	i	
i	i	i	i	i	j
i	į	į	į	į	İ
İ	1	1	1	1	
1	1	1	1	1	
		*******	*******		

LOW HIGH Acceptance Criteria: 85 125 Surrogate (n-Butanol) # Column to be used to flag recovery values

* Values outside QC limits

D Surrogates diluted out

Matrix Spike\Matrix Spike Duplicate

Selected VOCs on Charcoal Tubes

Spiked Sample Number : Spiked Dup Sample Number:

Batch Number: SDG Number:

Inj.: Inj.: Inj.:

Date :

:::::::::::::::::::::::::::::::::::::::			**********				*******
This MS/MSD	1 1	Spike	Sample	MS	MS	l ec	i
applies to the	i i	Added	Concentration	Concentration	*	Limits	Comments
following samples	Compound	(mg/mL)	(mg/mL)	(mg/mL)	REC	REC	i i
**************	****************	*******		*********		********	*******
1	Acetone	0.033	ND	0.033	100	75 -125	İ 1 1
i	Chlorobenzene	0.100	ND	0.100	100	75 -125	i i
i	Chloroform	0.100	ND ND	0.100	100	75 -125	i i
i	1,1-Dichloroethane	0.100	ND ND	0.100	100	75 -125	1 1
i	1,1-Dichloroethene	0.100	ND	0.100	100	75 -125	i i i
İ	Ethyl benzene	0.033	ND	0.033	100	75 -125	į į į
Ì		0.033	j ND j	0.033	100	75 -125	i i i
	Methyl isobutyl ketone	0.033	ND	0.033	100	75 -125	1 1
1	Methylene chloride	0.100	ND	0.100	100	75 -125	İ İ İ
İ	Tetrachloroethylene	0.100	ND	0.100	100	75 -125	
1	Toluene	0.033	ND	0.033	100	75 -125	İ İ İ
į	1,1,1-Trichloroethane	0.100	ND ND	0.100	100	75 -125	i i i
i	1,1,2-Trichloroethane	0.100	ND	0.100	100	75 -125	i i i
j	Trichloroethylene	0.100	ND	0.100	100	75 -125	i i
İ	Xylenes (sum of isomers)	0.033	ND	0.033	100	75 -125	i i

	Spike	MSD	MSD	90	: [ec
1	Added	Concentration	%	Limi	ts	Limit
Compound	(mg/mL)	(mg/mL)	REC	RE	C % RPD	RPD
Acetone	0.033	0.033	100	75 -1	25 0.0	1 15
Chlorobenzene	0.100	0.100	100	75 -1	25 0.0	15
Chloroform	0.100	0.100	100	75 -1	25 0.0	15
1,1-Dichloroethane	0.100	0.100	i 100	j75 -1	25 0.0	15
1,1-Dichloroethene	0.100	0.100	j 100	75 -1	25 0.0	j 15
Ethyl benzene	0.033	0.033	100	75 -1	25 0.0	15
· Methyl ethyl ketone	0.033	0.033	100	75 -1	25 0.0	15
Methyl isobutyl ketone	0.033	0.033	100	75 -1	25 0.0	j 15
Methylene chioride	0.100	0.100	100	75 -1	25 0.0	j 15
Tetrachloroethylene	0.100	0.100	100	j75 -1	25 0.0	j 15
Toluene	0.033	0.033	100	75 -1	25 0.0	į 15
1,1,1-Trichtoroethane	0.100	0.100	100	75 -1	25 0.0	j 15
1,1,2-Trichloroethane	0.100	0.100	100	75 -1	25 0.0	1 15
Trichloroethylene	0.100	0.100	100	75 -1	25 0.0	1 15
Xylenes (sum of isomers)	0.033	0.033	100	75 -1	25 0.0	j 15

Quality Control Summary Initial Calibration

Selected VOCs on Charcoal Tubes

Calibration Date....:

Instrument Identification..:

Laboratory Standard ID

	•	*********				========			EEEEEEEE
This IC applies		DF 2000	DF 400	DF 100	DF 40	DF 20	\	}	XRSD
to samples:	Compound	STD 1	STD 2	STD 3	STD 4	STD 5	AVE RF	XRSD	QC Limit
111111111111111111111111111111111111111	22822383222222222222222222222	*********		=======================================	========	======================================	********		********
1	Acetone	j	1	1	1	1)	l	25
	Chlorobenzene	1	i	I	1	l	l	l	25
1	Chloroform	1	1	1	l	l		l	25
	1,1-Dichloroethane	1	1	l	l	1	1]	25
	1,1-Dichloroethene	1	1	1	i	ì	1	1	25
	Ethyl benzene	İ	Ì	į.	İ	Ì	l	Ì	25
1	Methyl ethyl ketone	1	i		1	1	ĺ	1	25
	Methyl isobutyl ketone	1	ĺ	1	Ì	I	1	1	25
1	Methylene chloride	1	1	i	1	1	}	{	25
	Tetrachloroethylene	1	[1	1	1		ļ	25
	Toluene		1	1	1	1	1	1	25
1	1,1,1-Trichloroethane	1	1	1	1	1	}	1	25
	1,1,2-Trichloroethane	I	1	1	1	1	ŀ	1	25
1	Trichloroethylene	ı	1	1	l	1	l	!	25
	p-Xylene	j	1	1		Į	1	1	25
1	m-xylene	1	1	1	1	1	i	1	25
1	[o-Xyl ene	1	Į.	1	1	l	1	l	25
1 1	11	1	Ì	1	1	1		1	1
	11	ĺ	Ì	İ	İ	Ì	1	ĺ	1
	{ }	}	1	}			į	1	1
1	II]	1	j	1		!	1	1
	11	J.	i		[1	1	1 1
1	11	1	1	j	1	1	1	1	1

Quality Control Summary Continuing Calibration

Selected VOCs on charcoal tubes

Calibration Date....:

Instrument Identification..:

Inj #..:

Continuing Calibration Date:

Units: ug/mL

	=======================================		***********	*********	*=======
	Reference	Continuing	Acceptano		1
Compound	Concentration	Calib Result	85%-	115%	Out of Range
	***********	**********	***********		************
Acetone	100.000	}	85.0	115.0	1
Chlorobenzene	300.000	!	255.0	345.0	1
Chloroform	300.000	1	255.0	345.0	1
1,1-Dichloroethane	300.000	1	255.0	345.0	1
1,1-Dichloroethene	300.000	[255.0	345.0	1
Ethyl benzene	100.000	[85.0	115.0	1
Methyl ethyl ketone	100.000	1	85.0	115.0	1
Methyl isobutyl ketone	100.000	1	85.0	115.0	1
Methylene chloride	300.000	1	255.0	345.0	1
Tetrachloroethylene	300.000	1	255.0	345.0	1
Toluene	100.000	1	85.0	115.0	
1,1,1-Trichtoroethane	300.000	1	255.0	345.0	ļ
]]1,1,2-Trichloroethane	300.000	1	255.0	345.0	1
Trichloroethylene	300.000	1	255.0	345.0	
Xylenes	100.000	1	85.0	115.0	
11	}	1	i i]	Į
[]	1		[1	ſ
11	1	1]	
11	1		j i]	1
11	1				1
- 11					1
[]	1	1	!]	1
- 11	i	1	1		
- 11	1	1	[]	1
- 11	1	1	İ		l 1
- []	İ	İ			İ
II .	1	Ì	İ		1
ii ii	İ	İ	İ		Ì
		İ	İ		l j
	İ	İ	ĺ		l j
il .	İ	Ì	İ		Ì
11	j	İ	İ		İ
11	İ	İ	ĺ	ĺ	į
11	j	İ	İ	İ	i
11	İ	İ	İ	İ	i
• •	•	•	•	•	•

Matrix Spike\Matrix Spike Duplicate

Phenol on XAD-7 Tubes

Spiked Sample Number: Spiked Dup Sample Number: Batch Number: SDG Number: Inj.: Inj.: Inj.:

	D	a	t	e	
--	---	---	---	---	--

=======================================	=	************	********			======	========		=====
This MS/MSD applies to the following samples	1	Compound	Spike Added (mg/mL)		MS Concentration (mg/mL)	MS % REC	QC Limits REC	Comment	s
	Ī	Phenol	9.600	======================================	9.600	100	75 -125	***********	====:
į į	i		İ	j j	į		j		j
ļ ļ	!		ļ	!!!	ļ.		!		ļ
}	- {		}	!	ļ.		}		1
	ŀ		1				 		1
i	i		i	i i	i		! 		i
j j	i		i	i i	i		i		j
!	ļ		İ		ļ		ĺ		1
	ļ		1	!	ļ				!
	1		1		ļ		 		!
	i						1		{
i i	i		j .	j i	į		İ	•	İ
!	- 1		1	1 1	l		1		l
	=	***************		222222222222	*************		2222222		22222
	=		=======================================				**======		
i	1		Spike	I MSD 1		MSD	l ec	l ec i	

Compound	Spike Added (mg/mL)	MSD Concentration (mg/mL)	MSD % REC	QC Limits REC	 % RPD	QC Limit RPD
Phenol	9.600	9.600	100	75 -125	0.0	15
	İ	i • i	i	75 -125	İ	15
	ĺ	i i	i	75 -125	Ì	15
	İ	i i	İ	75 -125	İ	15
	İ	j j	İ	75 -125	İ	15
	j	İ	į	75 -125	İ	15
		İ		75 -125	İ	15
		1	1	75 -125	Ì	15
	1	1	1	75 -125	1	15
	ĺ	į į	j	75 -125	İ	15
		1	1	75 -125	j	15
	1	1	1	75 -125	ĺ	15
			1	75 -125	İ	15
		1	İ	75 -125	I	15
	1	i i	İ	75 -125	İ	15



Method Blank

Phenol by HPLC

*** BLANK INFORMATION ***

Matrix....: XAD-7 Tube

Batch Number....:
Injection number...:
Analysis date...:

Sample Info	ormation		İ	Blank Conta	mination Information		
LLI Sample No.	Client Designation	Analy Date	Time	CAS Number		Blank Result (mg)	LOQ (mg)
**********		======================================			:=====================================	ND ND	0.008
	<u> </u>	 		i I			
		[]]					
	i 1	i 1		i I		i i	İ
	 	<u> </u>					
	1	 -					
] [! !					1
	i 1						
	<u> </u> 		j 1 j	i 1		İ	j I
]] !]
]]]	} 					
	 	! ! !					
	İ	İ	j i	ii		j	i

Comments:

LOQ = Limit of Quantitation

ND = None Detected

NR = Not Reported

APPENDIX D

ATEC ASSOCIATES, INC.

STANDARD OPERATING PROCEDURES FOR PARTICLE SIZE DISTRIBUTION ANALYSIS

ATEC ASSOCIATES, INC. STANDARD OPERATING PROCEDURE PARTICLE SIZE DISTRIBUTION ANALYSIS

1. Parameter: Soil particle size distribution.

2. Range of Measurement: 0.0 - 100%

3. Limit of Detection: Not Applicable.

4. Sample Matrix: Soil.

- 5. Principle, Scope, and Application: Determine the particle size distribution in order to help classify the soil type and identify its properties.
- 6. Interferences and Corrective Actions: Equipment failure or technician errorreadjust, repair, or replace defective equipment and/or discuss error with the technician. Conduct duplicate test as necessary.
- 7. Safety Precautions: Wear safety glasses or gloves as needed for the contaminant present. Use alternate preparation methods if necessary.
- 8. Sample Size, Collection, Preservation, and Handling: Sample size is dependent upon the largest diameter sediment grain in the soil to be analyzed. Each sample should be delivered/shipped in a sealed container (bag, jar, etc.) that is of sufficient size to contain the sample and will not permit the loss of any portion of the sample.

- 9. Apparatus:
 - Balance sensitive to 0.01g,
 - Mortor and rubber-tipped pestle,
 - Stirring device "A" (per ASTM D-422; para. 3.2.1),
 - Sedimentation cylinder (1000 ml),
 - Soil Hydrometer 152-H (per ASTM D-422, para 3.3), and
 - Sieves (per ASTM D-422; para. 3.6).
- 10. Routine Preventative Maintenance: Balances are checked and serviced by a qualified service technician annually, and spot checked as needed. Sieves are checked with glass calibration spheres annually.
- 11. Reagents & Calibration Standards: None.
- 12. Calibration Procedures: None.
- 13. Sample Preparation: Samples are air dried at room temperature then broken down to particle size using a mortor and rubber-tipped pestle.
- 14. Analytical Measurement: Not Applicable.
- 15. Flow Chart: See Attached Exhibit.

16. Data Treatment:

% Soil in Suspension

 $P = (Ra/W) \times 100$

R = Hydrometer reading

a = Correction factor

(Table 1, ASTM D-422)

W = Oven dry soil weight

Diameter of Soil Particles

 $D = K \times SQRT (L/T)$

K = Constant (Table 3; ASTM

D-422)

L = Effective depth

(Table 2; ASTM D-422)

T = Elapse time of reading

SQRT = Square root

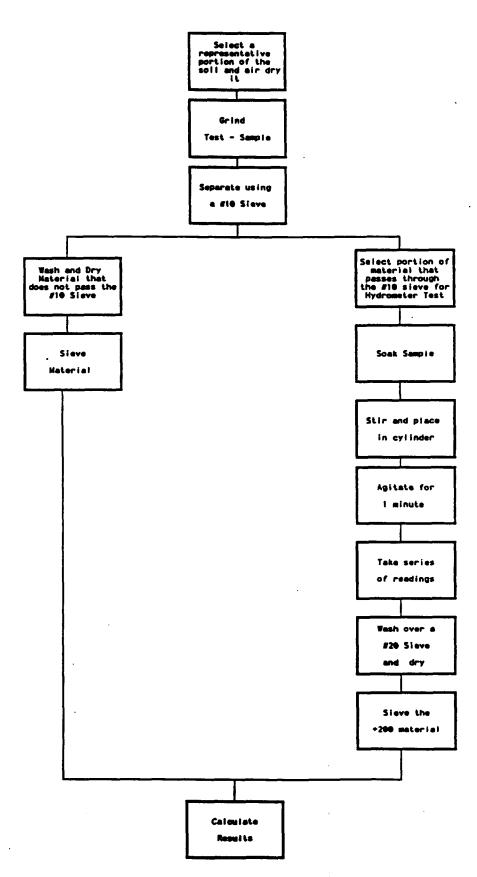
17. Data Deliverables:

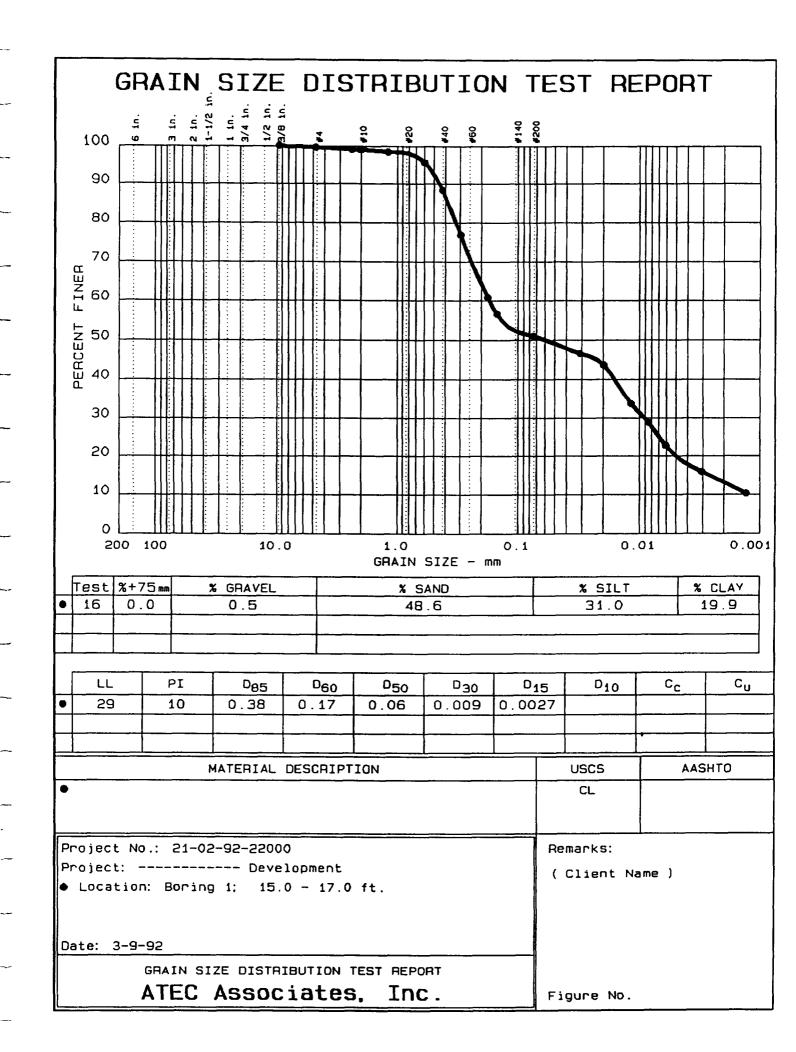
- a. Case narrative.
- b. Summary of all tests, and QC duplicate results (if required).
- c. Copies of lab data sheets (if required).
- d. Copies of any other pertinent sample documentation.

An example test report is attached.

- 18. Quality Control Requirement: Duplicate test as requested.
- 19. **References:** ASTM D-422-63.
- 20. Method Validation Data: Not Applicable.

Particle Size Analysis Flow Chart





```
GRAIN SIZE DISTRIBUTION TEST DATA
                                          Test No.: 16
3-9-92
Project No.: 21-02-92-22000 roject: Development
Sample Data
Location of Sample: Boring 1; 15.0 - 17.0 ft.
ample Description:
__SCS Class:
             CL
                          Liquid limit:
                                       29
                          Plasticity index: 10
AASHTO Class:
                        Notes
Remarks: ( Client Name )
⊸'ig. No.:
             Mechanical Analysis Data
......
           Initial
Ory sample and tare= 454.10
               25.40
Dry sample weight = 428.70
Sample split on number 10 sieve
Split sample data:
_ Sample and tare = 156.77 Tare = 107.41 Sample weight = 49.36
 Cumulative weight retained tare= 107.41
Tare for cumulative weight retained= 25.4
          Cumul. Wt. Percent
 Sieve
          retained
                  finer
           25.40
27.46
29.37
 0.375 inches
                   100.0
                   99.5
 # 4
-- # 8
                    99.1
 # 10
            29.88
                    99.0
      107.68 98.4

109.12 95.5

112.70 88.3

118.45 76.8

126.44 60.8

128.54 56.6

131.36 50.9
 # 16
# 30
 # 40
 # 50
 # 80
 # 100
              Hydrometer Analysis Data
```

reparation sieve is number 10

'ercent -# 10 based on complete sample= 99.0

Weight of hydrometer sample: 50

Hygroscopic moisture correction:

Moist weight & tare = 35.31

Dry weight & tare = 35.00
Tare = 11.66
Hygroscopic moisture= 1.3 %
alculated biased weight= 49.87
Automatic temperature correction
Composite correction at 20 deg C =-6.5

	Elapsed time, min		Actual reading	Corrected reading	K	Rm	Eff. depth	Diameter mm	Percent finer
	2.0	24.0	29.0	23.5	0.0128	29.0	11.5	0.0308	46.5
	5.0	24.0	27.5	22.0	0.0128	27.5	11.8	0.0197	43.5
	15.0	24.0	22.5	17.0	0.0128	22.5	12.6	0.0118	33.6
~~	30.0	24.0	20.0	14.5	0.0128	20.0	13.0	0.0084	28.7
	60.0	24.0	17.0	11.5	0.0128	17.0	13.5	0.0061	22.7
·	250.0	24.0	13.5	8.0	0.0128	13.5	14.1	0.0030	15.8
	1440.0	23.0	11.0	5.2	0.0130	11.0	14.5	0.0013	10.2

Fractional Components

% + 3 in. = 0.0 % GRAVEL = 0.5 % SAND = 48.6 % SILT = 31.0 % CLAY = 19.9

USS 0.38 D60= 0.172 D50= 0.058 D30= 0.0092 D15= 0.00269

ATEC ASSOCIATES, INC. SOILS LABORATORY LABORATORY SAMPLE CUSTODY

Upon receipt, the chain-of-custody (if possible) is signed by the lab supervisor, or technician, that receives the samples. samples are then logged, and assigned a consecutive sample log A work order is prepared for the sample, or group of samples, and the test(s) that are to be conducted on each sample is indicated. If a sample requires both physical and chemical tests, separate chemistry lab work prepared and order is representative portion of the sample is obtained and taken to the chemistry lab. All work orders, sample tags, lab sheets, etc. must have the appropriate sample log numbers on them. portions of the samples are maintained in the laboratory for 30 days after the test is completed unless other arrangements are made by the client.

JOB N	AME .													BIGNE							
COMP		DE			•																
BORING	SAMPLE	рертн	EXTRUDE	MC	DENSITY	WASHED SIEVE	HYDR.	-200	ā	CONSOL.	UNCONF.	.no	र्ड	FALLING HEAD	TRIAX.	PRO	CTOR Q Q	CBR	5	QΤΙ	HEA
	\vdash		<u> </u>	-								_	}						_		-
•																					
			 																	 	
	1-		-		 	_							 		<u> </u>						-
			F																<u> </u>		
~	-		-	-		-										-				-	├─
			-	-				<u> </u>				ļ	 			├					├
			├-	-		-					-	-									╁
																					Γ
			├-	-		-						-		} - · -		 	_				┼─
			ļ		 —	-					_	<u> </u>	 				 			-	├—
	+-		╁	┼─	-	-	\vdash	 			 		}						 —		
																				·	匚
	╁		-	 	 	-		<u> </u>		<u> </u>		-	 	┼		}		} -	 	-	├
	+		+-	-		-		-					-	1	- :-	-	 	_	_		
																					igspace
	+		╁╾	 	├	╁┈		├-	┧		 —	-	-	-	-	┼	 			┼╌	
	上					上															
		O. OF PTS.)							DUS N	MATE	RIALS	S	C) YE	is		10					
										<u></u>											



IOB NAME							DATE A					
	•											
		POSSIBLE	HAZARI	DOUS N	MATERIA	LS	☐ YES	Пи	0			
SAMPLE ((CIRCLE) SOIL AGGR BRICK FIRE OTHER:	PROOFING	ROC	FING	CEME	NT		TE COF	RES			
•	COMPUTER -											
	NOIT	PROCTOR	SIE	VE	N O		⊒ ≿	SSIVE	OTHER			
SAMPLE LOG NO.	SAMPLE SOURCE AND DESCRIPTION	STD	-200 ONLY	WASHED	EXTRACTION A GRADATION	DENSITY	MARSHAL STABILITY & FLOW	COMPRESSIVE STRENGTH				
							 					+
								· -		!		
			 		 - -		!					
					-							1.
			-		<u> </u>			·				-
_					-							
					:							-
SAMPLE LO	I DCATION: DLI INSTRUCTION		PROCT	L OR ROC) M : D	MAIN L	AB C	CONC	RETE LAI	3	· ·	
					· · · · · · · · · · · · · · · · · · ·							

MBH I NOIL	PALE LOCA	•	AT V	EG		Se Divis 5150	eion Eas	of A	C(TEC th St	Assor) ociate	es, io	ıc.					T	Deceived hv. (Stoneture)	SECTION REVISION PAGE	
		8		<u> </u>	-	_						1							1		one #:
(4)	AUTION TO		\dashv	-	+	+	\dashv	\dashv	\dashv		$\frac{1}{1}$		1	1	1	1					Jer / Ph
SNOBUL	94 /	S														1		 		nature)	Project Menager / Phone #:
	TOWN !	82														1	$\frac{1}{1}$	1		Relinquished by: (Signature)	Time
, т	10 40 37U N	18/25										.	_	1	<u> </u>	1	1	-	-	Relinquish	Date / Time
	.Q.I FE38	BAJ MUM									_		_	_	_	4	-	-	4		š
1	BER OF	CON	_	_	_	_					_			_	-	-	\dashv		-	Ignature)	boratory by:
7		g201	-		+	_								\dashv		7	\dashv	\dashv		(Signal	Labor
	03141	ACID			\dashv	-														ed by:	ed for
	Q3A3	3TJI3			1												_			Received by: (SI	Received for La (Signature)
					\dashv	_		_	-			-					_	-			
	н	SOIL			_{			_		T										Date / Time	Date / Time
		BARD	-																	Oate	Oate
	STIE	COME					_	<u> </u>	_	-	_	_	-								
		TIME							_	_		_		_						nature)	neture)
gnature)	1HOD	DATE												_		_	_			1 by: (Sigi	d by: (Sig
SAMPLERS: (Signature)	SAMPLING METHOD	SAMPLE I.D. NO.																		Relinquished by: (Signature)	Relinquished by: (Signeture)

SECTION NO.: 7

REVISION NO.: 0

FATE: 8/28/90

PAGE 6 OF 6

IN HOUSE CHAIR OF CUSTODY

EC LAB NO	CITEST IOR N	·n						
ATRIX								
ATRIX XIRACIION REQUIRED (Y OR K)	ORGANIC Y/NINORGANIC	¥/k						
AMPLES PLACED IN COOLER: DATE	E TIME by							
RELIKQUISHED BY:								
		·						
•		·						
		<u> </u>						
								
SAMPLES/EXTRACTS PLACED IN	POST AKALYSIS STORACE BY							
DATE								
SAMPLES/EXTRACTS DISPOSED C								
DATE	71HE							
NETHOD OF DISPOSAL								

APPENDIX E

IEA, INC.

STANDARD OPERATING PROCEDURE FOR ARSENIC AND ANTIMONY ANALYSIS AND OTHER INFORMATION

APPENDIX E

IEA, INC.

STANDARD OPERATING PROCEDURE FOR ARSENIC AND ANTIMONY ANALYSIS AND OTHER INFORMATION

TABLE OF CONTENTS

E.1	Standard Operating Procedure for Arsenic and Antimony Analysis
E.2	Sample Custody

- E.3 Data Reduction and Reporting
- E.4 Performance and System Audits
- E.5 Corrective Action

APPENDIX E.1

IEA, INC.

STANDARD OPERATING PROCEDURE FOR ARSENIC AND ANTIMONY ANALYSIS

STANDARD OPERATING PROCEDURE FOR ARSENIC AND ANTIMONY ANALYSIS

IEA, Inc. will be analyzing surface and subsurface waters collected from the Enviro-Chem site for arsenic and antimony using the EPA Method 200.8 "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectroscopy," a copy of which is included in this Appendix. Since the attached method is not a project specific SOP, this introduction has been created to provide laboratory- and project-specific items regarding this analysis.

1. Parameters to be Measured

Arsenic and antimony will be measured in subsurface and surface waters.

2. Range of Measurement

IEA has not yet completed a linear calibration range study. They expect to have completed it before samples are collected for this project, and will report their results to the water sampling contractor when they are available. The contractor will forward the results to the USEPA and IDEM.

3. Method Detection Limit (MDL)

IEA has achieved the following detection limits with this method: 1.2 ug/l for arsenic and 0.2 ug/l for antimony. Since the cleanup objective for arsenic is 0.0175 ug/l, the sample will be concentrated 100 times so that a theoretical MDL of 0.012 ug/l is achieved.

4. Sample Matrix

The matrices for this project are subsurface and surface waters.

5. Principle, Scope, and Application

Please refer to Section 1 of the method for this information.

6. Interferences and Corrective Actions

Please refer to Section 4 of the method for this information.

7. Safety Precautions

Please refer to Section 5 of the method for this information.

8. Sample Size, Collection, Preservation, and Handling

Two liters of sample will be collected in Teflon containers that are pre-weighed and labelled by the laboratory. IEA will obtain I-Chem 200 series bottles and send them to the sample collection personnel. The samples should be preserved in the field by adding ultra-pure nitric acid until the pH is less than 2. Subsurface water samples will be filtered using a 0.45 u non-metallic filter. All samples will be stored at 4°C. The holding time for these samples is 6 months from the date of collection.

9. Apparatus

A VG Plasmaquad ICP-MS is used with the following operating conditions:

- o 1250 watts
- o pulse counting mode
- o 0.75 1/min nebulizer flow rate
- o 3 integrations at 1 minute each
- o 3 internal standards (Sc, Ho, and In) at 100 ug/l each.

Also, an electron multiplier detector, as mentioned in Section 6.1.6, is used.

10. Routine Preventative Maintenance

The following are performed daily or whenever the instrument is used:

- o Tune instrument.
- o Check mass calibration.
- O Check the cone for wear (includes monitoring for Ba⁺⁺ and BaO), and replace if necessary (extra cones are kept on the premises).
- o Monitor vacuum settings.

Additionally, the detectors are changed whenever indicated by a drop in the intensity on In. Currently, this occurs about every 6 to 8 months at IEA. Whenever a detector is changed, the MDL study is re-done. IEA has a maintenance group that can perform many of the maintenance and repair tasks on this equipment. The laboratory also has a service contract with Fisions and will be able to get 24-hour turnaround on service by the time these samples are collected.

11. Reagents and Calibration Standards

The reagents are described in Section 7 of the method. The calibration levels to be used are as follows:

- o Instrument calibration 100 ug/l each of arsenic and antimony. A blank is also used for instrument calibration.
- o Initial calibration verification (ICV) arsenic at 200 ug/l, antimony at 120 ug/l.
- o Continuing calibration 100 ug/l each of arsenic and antimony.

The spiking solution to be used will contain arsenic and antimony at 2 times the MDL.

12. Calibration Procedures

Calibration is described in Section 9 of the method. The instrument calibration (using a blank and the standard described above) then the ICV are performed before analyzing any samples. The continuing calibration check is performed every 10 samples. If the

continuing calibration check results in a reading deviating by greater than 10% from the standard, the instrument will be recalibrated and the previous samples reanalyzed.

13. Sample Preparation

- o Record the weight of the sample, bottle, and label(s).
- o Loosen bottle cap and place sample in microwave.
- O Concentrate sample by evaporation. Check the sample periodically to avoid over concentrating. Use a reference bottle containing the desired final volume to aid in estimating the completion of the concentration. It takes approximately 3 hours to complete a 100:1 concentration of a 2-liter sample.
- o When concentration is complete, let sample cool, leaving the cap loosened.
- o Tighten cap.
- o Weigh concentrated sample, bottle, and label.
- o Calculate the concentration factor as follows:

```
<u>weight (pre-conc.) - empty bottle (with label)</u> = Conc. factor weight (post-conc.) - empty bottle (with label)
```

14. Analytical Measurement

Please refer to Sections 11.3 through 11.8.

15. Data Treatment

Please refer to Section 12.

16. Data Deliverables

The following items will be in the data package, which will be sent to the contractor:

- o Case narrative, briefly describing the sample preparation and analysis, problems encountered, and corrective actions taken.
- o Summary of initial and continuing calibrations.
- o Summary of sample analytical results.
- o Summary of QC sample analytical results.
- o Raw data, including instrument printouts.

17. Quality Control Requirements

The laboratory blanks will be analyzed at the rate of one per group of 10 or fewer samples. For both the laboratory and field blanks, the presence of elements at levels greater than the MDL will indicate laboratory or field contamination of the samples.

The acceptance criteria for duplicates is $\pm 20\%$.

Matrix spike samples (called "laboratory fortified sample matrix" in the method) will be prepared and analyzed for every group of 20 or fewer samples. The acceptance criteria for these samples is 85-115%.

18. References

Please refer to Section 14 of the method.

METHOD 200.8

ز إسامه لم

DETERMINATION OF TRACE ELEMENTS IN WATERS AND WASTES BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROSCOPY (Version 4.3).

August 1990

Stephen E. Long, Technology Applications Inc.; Theodore D. Martin, Chemistry Research Division; Environmental Monitoring and Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati,

	Or	710 45266.	
DEY		dissolved solids level should not exceed 0.2% (w/v), see	

_		2.0001100 00.00		,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,
	ction Subject Page /, /,	4.1.4,		
Nu	mber γ , γ	1.3 This metho	d is app	icable to the following elements:
1	Scope and Application Summary of Method	Element		Chemical Abstract Services
2	Summary of Method			Registry Numbers (CAS RN)
3	Definitions / //	Aluminum	(Al)	7429-90-5
4	Definitions Interferences AS 1.4	Antimony	(Sb)	7440-36-0
5	Salety 2 64	Arsenic	(As)	7440-38-2
6	Apparatus and Equipment > b	Barium	(Ba)	7440-39-3
7	Apparatus and Equipment Share Reagents and Consumable Materials	Beryllium	(Be)	7440-41-7
8	Sample Collection, Preservation and Storage	Cadmium	(Ca)	7440 <u>4</u> 3_9
9	Calibration and Standardization	Chromium	(Cr)	7440-47-3
10	Quality Control	Cobalt	(Co)	7440 <u>48-4</u>
11	Procedure	Copper	(Cu)	7440 <u>-50</u> -8
12	Calculations	Lead	(Pb)	7439-92-1
13	Precision and Accuracy	Manganese	(Mn)	7 439 –96–5
14	References	Molybdenum	(Mo)	7439-98-7
	TABLES	Nickel	(Ni)	7440-02-0
1.	Estimated Instrument Detection Limits, Method 200.8	Selenium	(Se)	<i>7782-49-2</i>
2.	Common Molecular Ion Interferences in ICP-MS.	Silver	(Ag)	7440-22-4
3.	Internal Standards and Limitations of Use.	Thallium	ĬΠ	7440-28-0
4.	Recommended Analytical Isotopes and Additional	Thorium	(Th)	7440 <u>-29</u> -1
	Masses which Must be Monitored.	Uranium	(U)	7440-61-1
5.	Recommended Elemental Equations for Data Calcu-	Vanadium	ίχί	7440-62-2

Zinc

Estimated instrument detection limits for these elements are listed in Table 1. These are intended as a guide to instrumental limits typical of a system optimized for multielement determinations and employing commercial instrumentation and pneumatic nebulization introduction. However, actual method detection limits and linear working ranges will be dependent on the sample matrix, instrumentation and selected operating conditions.

7440-66-6

(Zn)

- 1.4 This method is suitable for the determination of silver in aqueous samples containing concentrations up to 0.1 mg/L. For the analysis of wastewater samples containing higher concentrations of silver, succeeding smaller volume, well mixed sample aliquots must be prepared until the analysis solution contains <0.1 mg/L silver.
- 1.5 This method should be used by analysts experienced in the use of inductively coupled plasma-mass spectrometry (ICP-MS), the interpretation of spectral and matrix interferences and procedures for their correction. A minimum of six months experience with commercial instrumentation is recommended.

	INDEX /
Sec	tion Subject Page
Nur	mber Y, Y
1	Scope and Application / / / / /
2	Summary of Method
3	Definitions
4	stion Subject Page mber Scope and Application Summary of Method Definitions Interferences Safety Apparatus and Equipment Reagents and Consumable Materials Sample Collectice Processing and Steppes
5	Salety 2 64
6	Apparatus and Equipment
7	Reagents and Consumable Materials
8	Sample Collection, Preservation and Storage
9	Calibration and Standardization
10	
11	Procedure
12	Calculations
13	
14	References
	TABLES
4	Estimated Instrument Detection Limits, Method 200.8
٠.	reprinted incommittee paraction filling, Wallion 500'9

IM

- Instrument Operating Conditions for Precision and
- Accuracy Data.
- Total Recoverable Method Detection Limits.
- Precision and Recovery Data in Aqueous Matrices.
- Precision and Recovery Data in Solid Matrices.

METHOD 200.8 — DETERMINATION OF TRACE ELE-MENTS IN WATERS AND WASTES BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY.

I. SCOPE AND APPLICATION

- 1.1 This method provides procedures for the determination of dissolved elements in ground waters, surface waters and drinking water supplies. It may also be used for the determination of total recoverable element concentrations in these waters as well as wastewaters, sludges and solid waste samples.
- 1.2 Dissolved elements are determined after suitable filtration and acid preservation. Acid digestion procedures are required prior to the determination of total recoverable elements. In order to reduce potential interferences, the

ICP Information Newsletter, Volume 16, Number 8, 460 (January 1991)

2. SUMMARY OF METHOD

2.1 The method describes the multi-element determination of trace elements by ICP-MS14. Sample material in solution is introduced by pneumatic nebulization into a radiofrequency plasma where energy transfer processes cause desolvation, atomization and ionization. The lons are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their massto-charge ratio by a quadrupole mass spectrometer having a minimum resolution capability of 1 amu peak width at 5% peak height. The ions transmitted through the quadrupole are registered by a continuous dynode electron multiplier or Faraday detector and the ion information processed by a data handling system. Interferences relating to the technique (section 4) must be recognized and corrected for. Such corrections must include compensation for isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents or sample matrix. Instrumental drift as well as suppressions or enhancements of instrument response caused by the simple matrix must be corrected for by the use of Internal standardization.

3. DEFINITIONS

- 3.1 DISSOLVED Material that will pass through a 0.45 µm membrane filter assembly, prior to sample addification.
 3.2 TOTAL RECOVERABLE The concentration of analyte determined on an unfiltered sample following treatment with hot dilute mineral acid.
- 3.3 INSTRUMENT DETECTION LIMIT (IDL) The concentration equivalent of the analyte signal, which is equal to three times the standard deviation of the blank signal at the selected analytical mass(es).
- 3.4 METHOD DETECTION LIMIT (MDL) The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero and determined from analysis of a sample in a given matrix containing analyte.

 3.5 LINEAR DYNAMIC RANGE (LDR) The concentration range over which the analytical working curve remainslinear.
- 3.6 LABORATORY REAGENT BLANK (LRB) (preparation blank) An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or apparatus.
- 3.7 CALIBRATION BLANK A volume of ASTM type I water acidified with the same acid matrix as is present in the calibration standards.
- 3.8 INTERNAL STANDARD Pure Analyte(s) added to a solution in known amount(s) and used to measure the relative responses of other method analytes that are components of the same solution. The internal standard must be an analyte that is not a sample component.
- 3.9 STOCK STANDARD SOLUTION A concentrated solution containing one or more analytes prepared in the laboratory using assayed reference compounds or purchased from a reputable commercial source.

- 3.10 CALIBRATION STANDARD (CAL) A solution prepared from the stock standard solution(s) which is used to calibrate the instrument response with respect to analyte concentration.
- 3.11 TUNING SOLUTION A solution which is used to determine acceptable instrument performance prior to calibration and sample analyses.
- 3.12 LABORATORY FORTIFIED BLANK (LFB) An aliquot of reagent water to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the method is within accepted control limits.
- 3.13 LABORATORY FORTIFIED SAMPLE MATRIX (LFM)
 An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for the concentrations found.
- 3.14 QUALITY CONTROL SAMPLE (QCS) A solution-containing known concentrations of method analytes derived from externally prepared test materials. The QCS is obtained from a source external to the laboratory and is used to check laboratory performance.

4. INTERFERENCES

- 4.1 Several interference sources may cause inaccuracles in the determination of trace elements by ICP-MS. These
- 4.1.1 Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. All elements determined by this method have, at a minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use with this method (Table 4), only molybdenum-98 (nuthenium) and selenium-82 (krypton) have isobaric elemental interferences. If alternative analytical isotopes having higher natural abundance are selected in order to achieve greater sensitivity, an isobaric interference may occur. All data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest. A record of this correction process should be included with the report of the data. It should be noted that such corrections will only be as accurate as the accuracy of the isotope ratio used in the elemental equation for data calculations. Relevant isotope ratios and instrument bias factors should be established prior to the application of any corrections.
- 4.1.2 Abundance sensitivity is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The potential for these

interferences should be recognized and the spectrometer resolution adjusted to minimize them.

4.1.3 Isobaric polyatomic ion interferences — are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use. These ions are commonly formed in the plasma or interface system from support gases or sample components. Most of the common interferences have been identified, and these are listed in Table 2 together with the method elements affected. Such interferences must be recognized, and when they cannot be avoided by the selection of alternative analytical isotopes, appropriate corrections must be made to the data. Equations for the correction of data should be established at the time of the analytical run sequence as the polyatomic ion interferences will be highly dependent on the sample matrix and chosen instrument conditions.

4.1.4 Physical interferences — are associated with the physical processes which govern the transport of sample into the plasma, sample conversion processes in the plasma, and the transmission of ions through the plasma—mass spectrometer interface. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g. viscosity effects), at the point of aerosol formation and transport to the plasma (e.g. surface tension), or during excitation and ionization processes within the plasma itself.

High levels of dissolved solids in the sample may contribute deposits of material on the extraction and/or skimmer cones reducing the effective diameter of the orifices and therefore ion transmission. Dissolved solids levels not exceeding 0.2% (w/v) have been recommended to reduce such effects. Internal standardization may be effectively used to compensate for many physical interference effects. Internal standards should have similar analytical behavior to the elements being determined.

4.1.5 Memory interferences — result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the sampler and skimmer cones, and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples (see 7.6.3). The possibility of memory interferences should be recognized within an analytical run and sultable rinse times should be used to reduce them. The rinse times necessary for a particular element should be estimated prior to analysis. This may be achieved by aspirating a standard containing elements corresponding to ten times the upper end of the linear range for a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of ten of the method detection limit, should be noted. Memory interferences may also be assessed within an analytical run by using a minimum of three replicate inlegrations for data acquisition. If the integrated signal values drop consecutively, the analyst should be alerted to the possibility of a memory effect, and should examine the analyte concentration in the previous sample to identify if this was high. If a memory interference is suspected, the sample should be re-analyzed after a long rinse period.

5. SAFETY

5.1 The toxicity or carcinogenicity of reagents used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method^{5.8}. A reference file of material data handling sheets should also be available to all personnel involved in the chemical analysis.

5.2 Analytical plasma sources emit radiofrequency radiation in addition to intense UV radiation. Suitable precautions should be taken to protect personnel from such hazards.

6. APPARATUS AND EQUIPMENT

6.1 INDUCTIVELY COUPLED PLASMA-MASS SPEC-TROMETER

6.1.1 Instrument capable of scanning the mass range 5-250 amu with a minimum resolution capability of 1 amu peak width at 5% peak height. Instrument may be fitted with a conventional or extended dynamic range detection system. 6.1.2 Aroon gas supply (high-purity grade, 99.99%).

6.1.3 A variable-speed peristaltic pump is required for solution delivery to the nebulizer.

6.1.4 A mass—flow controller on the nebulizer gas supply is required. A water—cooled spray chamber may be of benefit in reducing some types of interferences (e.g. from polyat—omic oxide species).

6.1.5 Operating conditions — because of the diversity of instrument hardware, no detailed instrument operating conditions are provided. The analyst is advised to follow the recommended operating conditions provided by the manufacturer. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions satisfy the analytical requirements and to maintain quality control data verifying instrument performance and analytical results. Instrument operating conditions which were used to generate precision and accuracy data for this method (section 13) are included in Table 6.

6.1.6 If an electron multiplier detector is being used, precautions should be taken, where necessary, to prevent exposure to high ion counts. Otherwise changes in instrument response or damage to the multiplier may result. Samples having high concentrations of elements beyond the linear range and with isotopes falling within scanning windows should be diluted prior to analysis.

6.2 LABWARE — For the determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust, etc. A clean laboratory work area, designated for trace element sample handling must be used. Sample containers can introduce positive and negative errors in the determination of trace

elements by (1) contributing contaminants through surface desorption or leaching, (2) depleting element concentrations through adsorption processes. All re-usable labware (glass, quartz, polyethylene, Teffon etc.) Including the sample container should be cleaned prior to use. Labware should be soaked overnight and thoroughly washed with laboratory-grade detergent and water, rinsed with water, and soaked for four hours in a mixture of dilute nitric and hydrochloric acid (1+2+9), followed by rinsing with water, ASTM type I water and oven drying.

NOTE: chromic acid must not be used for the cleaning of glassware.

- 6.2.1 Glassware volumetric flasks and graduated cylinders.
- 6.2.2 Assorted calibrated pipettes.
- 6.2.3 Conical Phillips beakers, 250 mL with 50 mm watch glasses. Griffin beakers, 250 mL with 75 mm watch glasses. 6.2.4 Storage bottles — narrow mouth bottles, Tellon FEP (fluorinated ethylene propylene) with Teizel ETFE (ethylene tetrafluorethylene) screw closure, 125 mL and 250 mL capacities.
- 6.3 SAMPLE PROCESSING EQUIPMENT
- 6.3.1 Air Displacement Pipetter —digital pipet system capable of delivering volumes from 10 to 2500 microliters with an assortment of high quality disposable pipet tips.
- 6.3.2 Balance analytical, capable of accurately weighing to 0.1 mg.
- 6.3.3 Hot Plate (Corning PC100 or equivalent).
- 6.3.4 Centrifuge steel cabinet with guard bowl, electric timer and brake.
- 6.3.5 Drying oven gravity convection oven with thermostatic control capable of maintaining 105°C ± 5°C.

7. REAGENTS AND CONSUMABLE MATERIALS

- 7.1 Reagents may contain elemental impurities which might affect analytical data. Owing to the high sensitivity of ICP-MS, high-purity reagents should be used whenever possible. All acids used for this method must be ultra high-purity grade. Suitable acids are available from a number of manufacturers or may be prepared by sub-boiling distillation. Nitric acid is preferred for ICP-MS in order to minimize polyatomic ion interierences. Several polyatomic ion interferences result when hydrochloric acid is used (Table 2), however, it should be noted that hydrochloric acid is required to maintain stability in solutions containing antimony and sliver. When hydrochloric acid is used, corrections for the chloride polyatomic ion interferences must be applied to all data.
- 7.1.1 Nitric acid, concentrated (sp. gr. 1.41).
- 7.1.2 Nitric acid (1+1) add 500 mL conc. nitric acid to 1000 ml with ASTM type I water.
- 7.1.3 Nitric acid (1+9) add 100 mL conc. nitric acid to 1000 ml with ASTM type I water.
- 7.1.4 Hydrochloric acid, concentrated (sp. gr. 1.19).
- 7.1.5 Hydrochloric acid (1+1) add 500 mL conc. hydrochloric acid to 400 mL of ASTM type I water and dilute to 1
- 7.1.6 Hydrochloric acid (1+4) add 200 mL conc. hydrochloric acid to 1000 mL with ASTM type I water.
- 7.1.7 Ammonium hydroxide, concentrated (sp. gr. 0.902).

- 7.1.8 Tartaric acid (CAS RN 87-69-4).
- 7.2 WATER For all sample preparations and dilutions, ASTM type I water (ASTM D1193) is required. Suitable water may be prepared by passing distilled water through a mixed bed of anion and cation exchange resins.
- 7.3 STANDARD STOCK SOLUTIONS May be purchased from a reputable commercial source or prepared from ultra high-purity grade chemicals or metals (99.99-99.999% pure). All salts should be dried for one hour at 105 *C. unless otherwise specified. (CAUTION: Many metal saits are extremely toxic if inhaled or swallowed. Wash hands thoroughly before handling). Stock solutions should be stored in Tellon bottles. The following procedures may be used for preparing standard stock solutions:
- NOTE: Some metals, particularly those which form surface oxides, require cleaning prior to being weighed. This may be achieved by pickling the surface of the metal in acid. An amount in excess of the desired weight should be pickled repeatedly, rinsed with water, dried and weighed until the desired weight is achieved.
- 7.3.1 Aluminum solution, stock 1 mL = 1000 µg Al: Pickle aluminum metal warm (1+1) HCl to an exact weight of 0.100 g. Dissolve in 10 mL conc. HCl and 2 mL conc. nitric acid, heating to effect solution. Continue heating until volume is reduced to 4 mL. Cool and add 4 mL ASTM type I water. Heat until the volume is reduced to 2 mL. Cool and dilute to 100 mL with ASTM type I water.
- 7.3.2 Antimony solution, stock 1 mL = 1000 µg Sb: Dissolve 0.100 g antimony powder in 2 mL (1+1) nitric acid and 0.5 ml conc. hydrochloric acid, heating to effect solution. Cool, add 20 mL ASTM type I water and 0.15 g tartaric acid. Warm the solution to dissolve the white precipitate. Cool and dilute to 100 mL with ASTM type I water.
- 7.3.3 Arsenic solution, stock 1 mL = 1000 µg As: Dissolve 0.1320 g As,O, in a mixture of 50 ml ASTM type I water and 1 mL conc. ammonium hydroxide. Heat gently to dissolve. Cool and acidity the solution with 2 mL conc. nitric acid. Dilute to 100 mL with ASTM type I water.
- 7.3.4 Barium solution, stock 1 mL = 1000 µg Ba: Dissolve 0.1437 g BaCO, in a solution mixture of 10 mL ASTM type I water and 2 mL conc. nitric acid. Heat and stir to effect solution and degassing. Dilute to 100 mL with ASTM type
- 7.3.5 Beryllium solution, stock 1 mL = 1000 µg Be: Dissolve 1.965 g BeSO, 4H,O (DO NOT DRY) in 50 mL ASTM Type I water. Add 1 ml conc. nitric acid. Dilute to 100 ml with ASTM Type I water.
- 7.3.6 Bismuth solution, stock 1 mL = 1000 µg Bl: Dissolve 0.1115 g Bl.O. in 5 ml conc. nitric acid. Heat to effect solution. Cool and dilute to 100 mL with ASTM type I water. 7.3.7 Cadmium solution, stock 1 mL = 1000 µg Cd: Pickle cadmium metal in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water. 7.3.8 Chromium solution, stock 1 mL = 1000 µg Cr. Dissolve 0.1923 g CrO, in a solution mixture of 10 mL ASTM type I water and 1 mL conc. nitric acid. Dilute to 100 mL with ASTM type I water.
- 7.3.9 Cobalt solution, stock 1 mL = 1000 µg Co: Pickie cobalt metal in (1+9) nitric acid to an exact weight of 0.100

g. Dissolve in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water. 7.3.10 Copper solution, stock 1 mL = 1000 µg Cu: Pickle copper in metal. In (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 ml (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water. 7.3.11 Indium solution, stock 1 mL + 1000 µg In: Pickle indium metal in (1+1) nitric acid to an exact weight of 0.100 g. Dissolve in 10 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water. 7.3.12 Lead solution, stock 1 mL = 1000 µg Pb: Dissolve 0.1599 g PbNO₃ in 5 mL (1+1) nitric acid. Dilute to 100 mL with ASTM type I water.

7.3.13 Magnesium solution, stock 1 mL \approx 1000 µg Mg: Dissolve 0.1658 MgO in 10 ml (1+1) nitric acid, heating to effect solution. cool and dilute to 100 mL with ASTM type I water.

7.3.14 Manganese solution, stock 1 mL = 1000 µg Mn: Pickle manganese flakes in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 ml (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 ml with ASTM type I water.

7.3.15 Molybdenum solution, stock 1 mL = 1000 µg Mo: Dissolve 0.1500 g MoO₃ in a solution mixture of 10 mL ASTM type I water and 1 mL conc. ammonium hydroxide., heating to effect solution. Cool and dilute to 100 ml with ASTM type I water.

7.3.16 Nickel solution, stock 1 mL = 1000 µg Ni: Dissolve 0.100 g nickel powder in 5 mL conc. nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.

7.3.17 Scandium solution, stock 1 ml = 1000 µg Sc; Dissolve 0.1534 g Sc₂O₃ in 5 ml (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 ml with ASTM type I water.

7.3.18 Selenium solution, stock 1 mL = 1000 µg Se: Dissolve 0.1405g SeO₂ in 20 mL ASTM type I water. Dilute to 100 ml with ASTM type I water.

7.3.19 Silver solution, stock 1 ml. = 1000 µg Ag: Dissolve 0.100 g silver metal in 5 ml (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 ml, with ASTM type I water. Store in dark container.

7.3.20 Terbium solution, stock 1 mL = 1000 μ g Tb: Dissolve 0.1176 g Tb4O7 in 5 mL conc. nitric acid. heating to effect solution. Cool and dilute to 100 mL with ASTM type i water. 7.3.21 Thallium solution, stock 1 mL = 1000 μ g Ti: Dissolve 0.1303 g TiNO₃ in a solution mixture of 10 mL ASTM type I water and 1 mL conc. nitric acid. Dilute to 100 mL with ASTM type I water.

7.3.22 Thorium solution, stock 1 mL = 1000 µg Th: Dissolve 0.2380 g Th(NO₃), 4H₂O (DO NOT DRY) in 20 mL ASTM type I water. Dilute to 100 mL with ASTM type I water.

7.3.23 Uranium solution, stock 1 mL = 1000 μ g U: Dissolve 0.2110 g UO₂(NO₃)₂.6H₂O (DO NOT DRY) in 20 mL ASTM type I water and dilute to 100 mL with ASTM type I water. 7.3.24 Vanadium solution, stock 1 mL = 1000 μ g V: Pickle vanadium metal in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water. 7.3.25 Yttrium solution, stock 1 mL = 1000 μ g Y: Dissolve

٠,

......

0.1270 g Y_2O_3 in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 ml with ASTM type I water. 7.3.26 Zinc solution, stock 1 mL = 1000 μ g Zn: Pickle zinc metal in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with ASTM type I water.

7.4 MULTI-ELEMENT STOCK STANDARD SOLUTIONS
— Care must be taken in the preparation of multi-element stock standards that the elements are compatible and stable. Originating element stocks should be checked for the presence of impurities which might influence the accuracy of the standard. Freshly prepared standards should be transferred to acid cleaned, not previously used FEP fluorocarbon bottles for storage and monitored periodically for stability. The following combinations of elements are suggested:

Standard So	lution A	Standard Solution B
Aluminum	Manganese	Barium
· Antimony	Molybdenum	Silver
Arsenic	Nickel	
Beryilium	Selenium	
Cadmium	Thallium	
Chromium	Thorium	•
Cobalt	Uranium	•
Copper	Vanadium	•
Lead	Zinc	

Multi-element stock standard solutions A and B (1 mL = 10 µg) may be prepared by diluting 1 mL of each single element stock in the combination list to 100 mL with AS TM type I water containing 1% (v/v) nitric acid.

7.4.1 Preparation of calibration standards — fresh multielement calibration standards should be prepared every two weeks or as needed. Dilute each of the stock multielement standard solutions A and B to levels appropriate to the operating range of the instrument using ASTM type I water containing 1% (v/v) nitric acid. The element concentrations in the standards should be sufficiently high to produce good measurement precision and to accurately define the slope of the response curve. Concentrations of 200 µg/L are suggested. If the spiking procedure is being used (9.2.1), add internal standards (7.5) to the calibration standards and store in Tellon bottles. Calibration standards should be initially verified using a quality control sample (7.8).

7.5 INTERNAL STANDARDS STOCK SOLUTION, 1 mL = 100 µg. Dilute 10 mL of scandium, yttrium, indium, terbium and bismuth stock standards (7.3) to 100 mL with ASTM type I water and store in a Teflon bottle. Use this solution concentrate to spike blanks, calibration standards and samples, or dilute by an appropriate amount using 1% (v/v) nitric acid, if the internal standards are being added by peristaltic pump (9.2.2).

7.6 BLANKS — Three types of blanks are required for this method. A calibration blank is used to establish the analytical calibration curve, the laboratory reagent blank is used to assess possible contamination from the sample preparation procedure and to assess spectral background and the rinse blank is used to flush the instrument between samples in order to reduce memory interferences.

in order to reduce memory interferences.

7.6.1 Calibration blank — consists of 1% (v/v) nitric acid in ASTM type I water. If the direct addition procedure is being used (9.2.1) add internal standards.

7.6.2 Laboratory reagent blank (preparation blank) — must contain all the reagents in the same volumes as used in processing the samples. The preparation blank must be carried through the entire sample digestion and preparation scheme. If the direct addition procedure (9.2.1) is being used, add internal standards to the solution after the preparation is complete.

7.6.3 Rinse blank—consists of 2% (v/v) nltric acid in ASTM type I water.

7.7 TUNING SOLUTION—This solution is used for instrument tuning and mass calibration prior to analysis. The solution is prepared by mixing beryllium, magnesium, cobalt, indium and lead stock solutions (see 7.3) in 1% (v/v) nitric acid to produce a concentration of 100 µg/L of each element. Internal standards are not added to this solution.

7.8 QUALITY CONTROL SAMPLE — The quality control sample will be available from the Quality Assurance Branch EMSL-Cincinnati. Dilute an appropriate aliquot of analytes (concentrations not to exceed 1000 µg/L), in 1% (v/v) nitric acid. If the direct addition procedure (9.2.1) is being used, add internal standards after dilution, mix and store in a Telion bottle.

7.9 LABORATORY FORTIFIED BLANK—To an aliquot of reagent blank, add aliquots from multi-element stock standards A and B (7.4) to produce a final concentration of 200 µg/L for each analyte. The fortified blank must be carried through the entire sample digestion and preparation scheme. If the direct addition procedure (9.2.1) is being used, add internal standards to this solution after preparation has been completed.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Prior to the collection of the sample, consideration should be given to the type of data required so that appropriate preservation and pretreatment steps can be taken. Filtration, acid preservation etc. should be performed at the time of sample collection or as soon thereafter as practically possible.

8.2 For the determination of dissolved elements, the sample should be filtered through a 0.45 μ m membrane filter. Use a portion of the sample to rinse the filter assembly, discard and then collect the required volume of filtrate. Acidity the filtrate with (1+1) nitric acid immediately following filtration to a pH of less than 2.

8.3 For the determination of total recoverable elements in aqueous samples, acidify with (1+1) nitric acid at the time of collection to a pH of less than 2 (normally, 3 mL of [1+1] nitric acid per liter of sample is sufficient for most ambient and drinking water samples). The sample should not be filtered prior to analysis.

NOTE: Samples that cannot be acid preserved at the time of collection because of sampling limitations or transport restrictions, should be acidified with nitric acid to a pH of less than 2 upon receipt in the laboratory. Following acidification, the sample should be beld for sixteen hours before

withdrawing an allquot for sample processing.

8.4 Solid samples usually require no preservation prior to analysis other than storage at 4°C.

9: CALIBRATION AND STANDARDIZATION

9.1 CALIBRATION — Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed and is required periodically throughout sample analysis as dictated by results of continuing calibration checks. After initial calibration is successful, a calibration check is required at the beginning and end of each period during which analyses are performed, and at requisite intervals.

9.1.1 Initiate proper operating configuration of instrument and data system. Allow a period of not less than thirty minutes for the instrument to warm up. During this process conduct mass calibration and resolution checks using the tuning solution. Resolution at low mass is indicated by magnesium isotopes 24, 25, 26. Resolution at high mass is indicated by lead isotopes 206, 207, 208. For good performance adjust spectrometer resolution to produce a peak width of approximately 0.75 amu at 5% peak height. Adjust mass calibration if it has shifted by more than 0.1 amu from unit mass.

9.1.2 Instrument stability must be demonstrated by running the tuning solution (7.7) a minimum of five times with resulting relative standard deviations of absolute signals for all analytes of less than 5%.

9.1.2 Prior to initial calibration, set up proper instrument software routines for quantitative analysis. The instrument must be calibrated for the analytes to be determined using the calibration blank (7.6.1) and calibration standards A and B (7.4.1) prepared at one or more concentration levels. A minimum of three replicate integrations are required for data acquisition. Use the average of the integrations for Instrument calibration and data reporting.

9.1.3 The rinse blank should be used to flush the system between solution changes for blanks, standards and samples. Allow sufficient rinse time to remove traces of the previous sample or a minimum of one minute. Solutions should be aspirated for thirty seconds prior to the acquisition of data to allow equilibrium to be established.

9.2 INTERNAL STANDARDIZATION — Internal standardization must be used in all analyses to correct for instrument drift and physical interferences. A list of acceptable internal standards is provided in Table 3. For full mass range scans, a minimum of three internal standards must be used. Procedures described in this method for general application, detail the use of five internal standards; scandium, yttrium, indium, terbium and bismuth. These were used to generate the precision and recovery data attached to this method. Internal standards must be present in all samples, standards and blanks at identical levels. This may be achieved by adding an aliquot of internal standards to the solution (method 9.2.1), or alternatively by mixing with the solution prior to nebulization using a second channel of the peristaltic pump and a mixing coil (method 9.2.2). The concentration of the internal standard should be sufficiently high that good precision is obtained in the measurement of the isotope used for data correction and to minimize the

possibility of correction errors if the internal standard is naturally present in the sample. A concentration of 200 µg/L of each internal standard is recommended. Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

9.3 INSTRUMENT PERFORMANCE — Check the performance of the instrument and verify the calibration using data gathered from analyses of calibration blanks, calibration standards and the quality control sample.

9.3.1 After the calibration has been established, it must be initially verified for all analytes by analyzing the QCS (7.8). If measurements exceed ±10% of the established QCS values, the analysis should be terminated, the source of the problem identified and corrected, the instrument re-calibrated and the calibration re-verified before continuing analyses.

9.3.2 To verify that the instrument is properly calibrated on a continuing basis, run the calibration blank and calibration standards as surrogate samples after every ten analyses. The results of the analyses of the standards will indicate whether the calibration remains valid. If the indicated concentration of any analyte deviates from the true concentration by more than 10%, reanalyze the standard. If the analyte is again outside the 10% limit, the instrument must be recalibrated and the previous ten samples re—analyzed. The instrument responses from the calibration check may be used for recalibration purposes. If the sample matrix is responsible for the calibration drift, it is recommended that the previous ten samples are re—analyzed in groups of five between calibration checks to prevent a similar drift situation from occurring.

10. QUALITY CONTROL

10.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, and the analysis of laboratory reagent blanks, fortified blanks and samples as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data thus generated.

10.2 INITIAL DEMONSTRATION OF PERFORMANCE.
10.2.1 The initial demonstration of performance is used to characterize instrument performance (method detection limits and linear calibration ranges) for analyses conducted by this method.

10.2.2 Method detection limits (MDL) — method detection limits should be established for all analytes, using reagent water (blank) fortified at a concentration of two to five times the estimated detection limit?. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

 $MDL = (t) \times (S)$

where, t = students' t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates].

S = standard deviation of the replicate analyses.

Method detection limits should be determined every six months or whenever a significant change in background or instrument response is expected (e.g. detector change). 10.2.3 Linear calibration ranges — linear calibration ranges are primarily detector limited. The upper limit of the linear calibration range should be established for each analyte by determining the signal responses from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range. Care should be taken to avoid potential damage to the detector during this process. The linear calibration range which may be used for the analysis of samples should be judged by the analyst from the resulting data. Linear calibration ranges should be determined every six months or whenever a significant change in instrument response is expected (e.g. detector change).

10.3 ASSESSING LABORATORY PERFORMANCE — REAGENT AND FORTIFIED BLANKS

10.3.1 Laboratory reagent blank (LRB) — the laboratory must analyze at least one reagent blank (7.6.2) with each set of samples. Reagent blank data are used to assess contamination from the laboratory environment and to characterize spectral background from the reagents used in sample processing. If an analyte value in the reagent blank exceeds its determined MDL, then laboratory or reagent contamination should be suspected. Any determined source of contamination should be corrected and the samples re-analyzed.

10.3.2 Laboratory fortified blank (LFB) — the laboratory must analyze at least one fortified blank (7.9) with each batch of samples. Calculate accuracy as percent recovery (see 10.4.2). If the recovery of any analyte falls outside the control limits (see 10.3.3), that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

10.3.3 Until sufficient data become available from within their own laboratory (usually a minimum of twenty to thirty analyses), the laboratory should assess laboratory performance against recovery limits of 85 – 115%. When sufficient internal performance data becomes available, develop control limits from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data are used to establish upper and lower control limits as follows:

UPPER CONTROL LIMIT = x + 3S LOWER CONTROL LIMIT = x - 3S

After each five to ten new recovery measurements, new control limits should be calculated using only the most recent twenty to thirty data points.

10.4 ASSESSING ANALYTE RECOVERY — LABORA-TORY FORTIFIED SAMPLE MATRIX

10.4.1 The laboratory must add a known amount of analyte to a minimum of 10% of the routine samples or one sample per set, whichever is greater. Ideally for water samples, the analyte concentration should be the same as that used in the laboratory fortified blank (10.3.2). For solid samples, the concentration added should be 50 mg/kg equivalent (100 µg/L in the analysis solution). Over time, samples from all routine sample sources should be fortified.

10.4.2 Calculate the percent recovery for each analyte, corrected for background concentrations measured in the unfortified sample, and compare these values to the control limits established in section 10.3.3 for the analyses of LFBs. Recovery calculations are not required if the concentration of the analyteis less than 10% of the sample background concentration. Percent recovery may be calculated in units appropriate to the matrix, using the following equation:

 $R = (C_a - C)100/s$

where, R = percent recovery.

C_s = fortified sample concentration.

C = sample background concentration.

s = concentration equivalent of fortifier added to sample. 10.4.3 If the recovery of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control (section 10.3), the recovery problem encountered with the fortified sample is judged to be matrix related, not system related. The result for that analyte in the unspiked sample must be labelled "suspect/matrix" to inform the data user that the results are suspect due to matrix effects.

10.5 INTERNAL STANDARDS RESPONSES.

The analyst is expected to monitor the responses from the internal standards throughout the sample set being analyzed. Ratios of the internal standards responses against each other should be monitored routinely. This information may be used to detect potential problems caused by mass dependent drift, errors incurred in solution spiking or increases in the concentration of individual internal standards caused by background contributions from the sample. The absolute response of any one internal standard should not deviate more than 60–125% of the original response in the calibration blank. If deviations greater than this are observed, use the following test procedure:

10.5.1 Flush the instrument with the rinse blank and monitor the responses in the calibration blank. If the responses of the internal standards are now within the limit, take a fresh aliquot of the sample, dilute by a further factor of two, respike with internal standards and re-analyze.

10.5.2 If test 10.5.1 is not satisfied, or if it is a blank or calibration standard that is out of limits, terminate the analysis, and determine the cause of the drift. Possible causes of drift may be partially blocked sampling cone or a change in the tuning condition of the instrument.

11. PROCEDURE

11.1 SAMPLE PREPARATION - DISSOLVED ELE-MENTS.

11.1.1 For the determination of dissolved elements in drinking water, ground and surface waters, take a 100 mL aliquot of the filtered acid preserved sample, and add 1 mL of concentrated nitric acid. If the spiking procedure (9.2.1) is being used, add internal standards and mix. The sample is now ready for analysis. Allowance for sample dilution should be made in the calculations.

Note: If a precipitate is formed during acidification, transport or storage, the sample aliquot must be treated using the procedure in section 11.2.1 prior to analysis.

11.2 SAMPLE PREPARATION — TOTAL RECOVERABLE ELEMENTS.

11.2.1 For the determination of total recoverable elements in water or wastewater, take a 100 mL aliquot from a well mixed, acid preserved, sample containing not more than 0.25% (w/v) total solids and transfer to a 250 ml. Griffin beaker. (If total solids are greater than 0.25% reduce the size of the aliquot by a proportionate amount). Add 1 mL of conc. nitric acid and 0.5 mL conc. hydrochloric acid. Heat on a hot plate at 85°C until the volume has been reduced to approximately 20 mL, ensuring that the sample does not boll. A spare beaker containing approximately 20 mL of water can be used as a gauge. (NOTE: Adjust the temperature control of hot plate such that an uncovered beaker containing 50 mL of water located in the center of the hot plate can be maintained at a temperature no higher than 85°C. Evaporation time for 100 mL of sample at 85°C is approximately two hours with the rate of evaporation increasing rapidly as the sample volume approaches 20 mL). Cover the beaker with a watch glass and reflux for thirty minutes. Slight bolling may occur but vigorous bolling should be avoided. Allow to cool and transfer to a 50 mL volumetric flask or 50 mL class A stoppered graduated cylinder. Dilute to volume with ASTM type I water and mix. Centrifuge the sample or allow to stand overnight to separate insoluble material. Prior to analysis; pipette 20 mL into a 50 mL volumetric flask, dilute to volume with ASTM type I water and mix. If the direct addition procedure (9.2.1) is being used, add internal standards and mix. The sample is now ready for analysis. Because the stability of diluted samples cannot be fully characterized, all analyses should be performed as soon as possible after the completed preparation.

11.2.2 For the determination of total recoverable elements in solid samples (sludge, soils, sediments), mix the sample thoroughly to achieve homogeneity and weigh accurately a 1.0 ± 0.01 g portion of the sample. Transfer to a 250 mL Phillips beaker. Add 4 mL (1+1) nitric acid and 10 mL (1+4) HCI. Cover with a watch glass, and reflux the sample on a hot plate for thirty minutes. Very slight boiling may occur, however, vigorous boiling must be avoided to prevent the loss of the HCI azeotrope. (NOTE: Adjust the temperature control of the hot plate such that an uncovered beaker containing 50 mL of water located in the center of the hot plate can be maintained at a temperature no greater than 85°C). Allow the sample to cool, and quantitatively transfer to a 100 mL volumetric flask. Dilute to volume with ASTM type I water and mix. Centrifuge the sample or allow to stand overnight to separate insoluble material. Prior to analysis, pipette 10 mL into a 50 mL volumetric flask and dilute to volume with ASTM type I water. If the direct addition procedure (9.2.1) is being used, add internal standards and mix. The sample is now ready for analysis. Because the effects of various matrices on the stability of diluted samples cannot be characterized, all analyses should be performed as soon as possible after the completed preparation.

NOTE: Determine the percent solids in the sample for use in calculations and for reporting data on a dry weight hasis

11.3 For every new or unusual matrix, it is highly recommended that a semi-quantitative analysis be carried out to screen for high element concentrations. Information gained from this may be used to prevent potential damage to the detector during sample analysis and to identify elements which may be higher than the linear range. Matrix screening may be carried out by using intelligent software, if available, or by diluting the sample by a factor of 500 and analyzing in a semi-quantitative mode. The sample should also be screened for background levels of all elements chosen for use as internal standards in order to prevent blas in the calculation of the analytical data.

11.4 Initiate instrument operating configuration. Tune and calibrate the instrument for the analytes of interest (see section 9).

11.5 Establish instrument software run procedures for quantitative analysis. For all sample analyses, a minimum of three replicate integrations are required for data acquisition. Discard any integrations which are considered to be statistical outliers and use the average of the integrations for data reporting.

11.6 All masses which might affect data quality must be monitored during the analytical run. As a minimum, those masses prescribed in Table 4 must be monitored in the same scan as is used for the collection of the data. This information should be used to correct the data for identified interferences.

11.7 The rinse blank should be used to flush the system between samples. Allow sufficient time to remove traces of the previous sample or a minimum of one minute. Samples should be aspirated for thirty seconds prior to the collection of data.

11.8 Samples having concentrations higher than the established linear dynamic range should be diluted into range and re-analyzed. The sample should first be analyzed for the trace elements in the sample, protecting the detector from the high concentration elements, if necessary, by the selection of appropriate scanning windows. The sample should then be diluted for the determination of the remaining elements. Alternatively, the dynamic range may be adjusted by selecting an alternative isotope of lower natural abundance, provided quality control data for that isotope have been established. The dynamic range must not be adjusted by altering instrument conditions to an uncharacterized state.

12. CALCULATIONS

12.1 Elemental equations recommended for sample data calculations are listed in Table 5. Sample data should be reported in units of µg/L for aqueous samples or mg/kg dry weight for solid samples. Do not report element concentrations below the determined MDL.

12.2 For data values less than ten, two significant figures should be used for reporting element concentrations. For data values greater than or equal to ten, three significant figures should be used.

12.3 Reported values should be calibration blank subtracted. For aqueous samples prepared by total recoverable procedure 11.2.1, multiply solution concentrations by the dilution factor 1.25. For solid samples prepared by total recoverable procedure 11.2.2, multiply solution concentrations (µg/L in the analysis solution) by the dilution factor 0.5. If additional dilutions were made to any samples, the

appropriate factor should be applied to the calculated sample concentrations.

12.4 Data values should be corrected for instrument drift or sample matrix induced interferences by the application of internal standardization. Corrections for characterized spectral interferences should be applied to the data. Chloride interference correction should be made on all samples, regardless of the addition of hydrochloric acid, as the chloride ion is a common constituent of environmental samples.

12.5 If an element has more than one monitored isotope, examination of the concentrations calculated for each isotope, or the isotope ratios, will provide useful information for the analyst in detecting a possible spectral interference. Consideration should therefore be given to both primary and secondary isotopes in the evaluation of the element concentration. In some cases, the secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes, therefore differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes.

12.6 The QC data obtained during the analyses provide an indication of the quality of the sample data and should be provided with the sample results.

13. PRECISION AND ACCURACY

13.1 Instrument operating conditions used for single laboratory testing of the method are summarized in Table 5. Total recoverable method detection limits determined using the procedure described in 10.2.2, are listed in Table 7. 13.2 Data obtained from single laboratory testing of the method are summarized in Table 8 for five water samples representing drinking water, surface water, ground water and waste effluent. Samples were prepared using the procedure described in 11.2.1. For each matrix, five replicates were analyzed and the average of the replicates used for determining the sample background concentration for each element. Two further pairs of duplicates were fortified at different concentration levels. For each method element, the sample background concentration, mean spike percent recovery, the standard deviation of the percent recovery and the relative percent difference between the duplicate fortified samples are listed in Table 8.

13.3 Data obtained from single laboratory testing of the method are summarized in Table 9 for three solid samples consisting of SRM 1645 River Sediment, EPA Hazardous Soil and EPA Electroplating Sludge. Samples were prepared using the procedure described in 11.2.2. For each method element, the sample background concentration, mean percent recovery, the standard deviation of the percent recovery and the relative percent difference between the duplicate spikes were determined as for 13.2.

14. REFERENCES

- 1. A.L. Gray and A.R. Date, Analyst 108 1033 (1983).
- 2. R.S. Houk et al. Anal. Chem. 52 2283 (1980).
- 3. R.S. Houk, Anal. Chem. 58 97A (1986).
- 4. J.J. Thompson and R.S. Houk, Appl. Spec. 41 801 (1987).
- 5. "OSHA Safety and Health Standards, General Industry,"

stration, OSHA 2206, revised January 1976.

- Proposed OSHA Safety and Health Standards, Laboratories, Occupational Safety and Health Administration, Federal Register, July 24, 1986.
- 7. Code of Federal Regulations 40, Ch. 1, Pt. 136 Appendix B.

NOTICE

This method has been peer and administratively reviewed by the Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

ACKNOWLEDGMENT

The authors gratefully acknowledge Eleanor Martin of the USEPA Senior Environmental Employment Program, Cincinnati, OH, for her assistance with sample preparation and comparative analyses using ICP-Atomic Emission Spectrometry, and Lisa Gragg, Computer Sciences Corporation, Cincinnati, OH, for guidance with the statistical interpretation of the data.

Special acknowledgement is accorded to the following individuals for their assistance with manuscript review: Larry Lobring, EMSL, Cincinnati, OH, Dr. Robert Hutton, VG Elemental, Winstord, Cheshire, UK, Dr. Raimund Roehl, California Public Health Foundation, Berkeley, CA, John Garbarino, US Geological Survey, Denver, CO, Thomas Hinners, Quality Assurance and Methods Development Division, EMSL, Las Vegas, NV and Guy Laing, Lockheed Engineering and Science Co., Las Vegas, NV.

TABLE 1. ESTIMATED INSTRUMENT DETECTION-LIMITS METHOD 200.8

ELEMENT	RECOMMENDED ANALYTICAL MASS	ESTIMATED IDL (µg/L)
Aluminum	27	0.05
Antimony	121	80.0
Arsenic	75	0.9
Barium	137	0.5
Beryllium	9	0.1
Cadmium	111	0.1
Chromium	52	0.07
Cobalt	59	0.03
Copper	63	0.03
Lead	206,207,208	80.0
Manganese	55	0.1
Molybdenu	m 98	0.1
Nickel	. 60	0.2
Selenium	82	5
Silver	107	0.05
Thallium	205	0.09
Thorium	232	0.03
Uranium	238	0.02
Vanadium	51	0.02
Zinc	66	0.2
instrument	detection limits (3s) estimated	from seven reolicate

instrument detection limits (3s) estimated from seven replicate scans of the blank (1% v/v nitric acid) and three replicate integrations of a multi-element standard.

TAE	BLE 2.	COMA	MON	MOLE	CULAR	ION
INT	ERFE	RENCE	ES IN	ICP-N	AS	
					AR ION	S

BACKGHOUN			
Molecular Ion	Mass	Element Interference	•
NH+ ·		15	
OH-		17	
OH,•		18	
C,·		24	
C. CN		26	
CO+		28 ·	
N,*		28	
N j H•		29	
NO-		30	
NOH-		31	
O*		32	
O'H.		33	
≃ ArH•		37	
™ArH•		39	
ArH		41	
co,•		44	
CO,H*		45	Sc
Arc+,ArO+		52	Cr
ArN-		54	Cr
AMH.		55	Mn
ArO-		56 .	
ArOH•		57	
ω\r _∞ \r _∞ \r		76	Se
"AISAA"		78	Se
4Ar2+		80	Se
-			

"Method elements or internal standards affected by the molecular ions.

TABLE 2 (Continued). MATRIX MOLECULAR IONS CHI ORIDE

	CHLORI	UE			
Molecular ion	Mass	Element Interference			
≈ C1O•	51	V			
≈CIOH•	52	Cr			
12(O-	53	Cr			
*COH	54	Cr			
AresCI+	75	As			
Araci.	77	Se			
	SULPHA	TE			
Molecular Ion	Mass	Element Interference			
320.	48				
#SOH+	49				
* \$0•	50	V, Cr			
¥SOH•	51	V			
SO, , S, .	64	Zn			
Arts.	72				
Aras.	74				
PHOSPHATE					
Molecular ion	Mass	Element Interference			
PO-	47				
POH+	48				
PO.	63	Cu			
ArP-	71				
GROUP I, II METALS					
Molecular ion	Mass	Element Interference			
ArNa•	63	Cu			

79

80

ArK.

ArCa+

ArCa•	80	
	MATRIX OX	(IDES*
Molecular Ion	Masses	Element Interference
TIO	62-66	Ni,Cu,Zn
ZO	106-112	Ag,Cd
MoO	108-116	Čď

*Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that TI and Zr isotopes are monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method analyte.

TABLE 3. INTERNAL STANDARDS AND LIMITATIONS OF USE

		110 01 000
internal standard	Mass	Possible Limitation
*Lithium	6	a
Scandium	45	polyatomic ion interference
Yttrium	89	a, b
Rhodium	103	
Indium	115	isobaric interference by Sn
Terbium	159	
Holmium	165	
Lutetium	175	<u>.</u>
Bismuth	209	a

- *May be present in environmental samples.
- In some instruments Yttrium may form measurable amounts of YO* (105 amu) and YOH* (106 amu). If this is the case, care should be taken in the use of the cadmium elemental correction equation.

Internal standards recommended for use with this method are shown in bold face. Preparation procedures for these are included in section 7.3.

TABLE 4. RECOMMENDED ANALYTICAL ISOTOPES AND ADDITIONAL MASSES WHICH MUST RE MONITORED

	MONITORED
isotope	Element of Interest
27	Aluminum
121,123	Antimony
75	Arsenic
1 35,13 7	Barium
2	Beryllium
106,108,111,114	Cadmium
52,53	Chromium
<u>59</u>	Cobalt
63 ,65	Copper
<u>206,207,208</u>	Lead
<u>55</u>	Manganese
95,97 <u>,98</u>	Molybdenum
60,62	Nickel
77,82	Selenium
107,109	Silver
203,205	Thallium
232	Thorium
238	Uranium
51	Vanadium
<u>66,67,68</u>	- Zinc

83	Krypton
99	Ruthenium
105	Palladium
118	Tin

NOTE: Isotopes recommended for analytical determination are underlined.

TABLE 5. RECOMMENDED ELEMENTAL EQUATIONS FOR DATA CALCULATIONS

Florent	Elemental Equation	خاملة
Element	•	Note
AI	(1.000)(°C)	
Sb	(1.000)(¹²¹ C)	
As	(1.000)(°C)-(3.127)[(°C)-(0.815)(°C)]	(1)
Ва	(1.000)([™] C)	
Be	(1.000)(°C)	
Cd	(1.000)(""C)-(1.073)[("C)-(0.712)("C)	(2)
Cr	(1.000)(^{sa} C)	(3)
Co	(1.000)(°C)	
Cu	(1.000)(°C)	
Pb	(1.000)(²⁰⁶ C)+(1.000)(²⁰⁷ C)+(1.000)(²⁰⁰ C)	(4)
Mn	(1.000)(⁴⁴ C)	
Mo	(1.000)(°C)-(0.146)(°C)	(5)
Ni	(1.000)(°C)	
Se	(1.000)(° C)	
Ag .	(1.000)(¹⁶⁷ C)	
ΤĬ	(1.000)(²⁰⁶ C)	
Th	(1.000)(™ C)	
U	(1.000)(²³ C)	
٧	(1.000)(51C)-(3.127)((5C)-(0.113)(5C))	(7)
Zn	(1.000)(*C)	
Bi	(1.000)(²⁰⁰ C)	
In	(1.000)(¹¹⁵ C)-(0.016)(¹¹⁸ C)	(8)
Sc	(1.000)(< C)	
Tb	(1.000)(159C)	
Y	(1.000)(°C)	
	(= -)(-)	

- C calibration blank subtracted counts at specified mass.
- (1) correction for chloride interference with adjustment for Se77. ArCl 75/77 ratio may be determined from the reagent blank.
- (2) correction for MoO interference. An additional isobaric elemental correction should be made if patientium is present.
- (3)—in 0.4% v/v HCl, the background from ClOH will normally be small. However the contribution may be estimated from the reagent blank.
- (4) allowance for isotope variability of lead isotopes.
- (5) isobaric elemental correction for ruthenium.
- (6) some argon supplies contain krypton as an impurity. Selenium is corrected for Kr82 by background subtraction.
- (7) correction for chloride interference with adjustment for Cr53. ClO 51/53 ratio may be determined from the reagent blank.
- (8) Isobaric elemental correction for tin.

TABLE 6. INSTRUMENT OPERATING CONDITIONS FOR PRECISION AND ACCURACY DATA

ID ACCURACY DATA
VG PlasmaQuad Type I
1.35 kW
13.5 L/min.
0.6 L/min .
0.78 L/min .
0.6 mL/min.

Spray chamber temperature	15 ° C
Data Acquisition	
Detector mode	Pulse counting
Replicate integrations	3
Mass range	8-240 amu
Dwell time	320 µs
Number of MCA channels	2048
Number of scan sweeps	85
Total acquisition time	3 minutes per sample

Th	<0.1	10	109.0	0.7	1.8	100	106.0	1.4	3.8			
U	0.23	10	110.7	1.4	3.5	100	107.8	0.7	1.9			
V	<2.5	50	101.4	0.1	0.4	200	97.5	0.7	2.1			
Zn	5.2	50	103.4	3.3	7.7	200	96.4	0.5	1.0			
S(R) Standard deviation of percent recovery.												
RPD Relative percent difference between duplicate spike deter-												
mina	tions.											

< Sample concentration below established method detection limit.</p>
*Spike concentration <10% of sample background concentration.</p>

TABLE 7. TOTAL RECOVERABLE METHOD DETECTION LIMITS

	DETECTION LIN	1115	
ELEMENT	RECOMMENDED	N	EDL.
•	ANALYTICAL MASS	AQUEOUS	SOLIDS
		µg/L	mg/kg
Aluminum	27	1.0	0.4
Antimony	121	0.4	0.2
Arsenic	75	1.4	0.6
Barium	137	0.8	0.4
Beryllium	9	0.3	0.1
Cadmium	111	0.5	0.2
Chromium	52	0.9	0.4
Cobalt	59	0.09	0.04
Copper	63	0.5	0.2
	206,207,208	0.6	0.3
Manganese	55	0.1	0.05
Molybdenum	n 98	0.3	0.1
Nickel	60	0.5	0.2
Seienium	82	7.9	3.2
Silver	107	0.1	0.05
Thallium	205	0.3	0.1
Thorium	232	0.1	0.05
Uranium	238 .	0.1	0.05
Vanadium	51	2.5	1.0
Zinc	66	1.8	0.7
1101			*** #

MDL concentrations are computed for original matrix with allowance for sample dilution during preparation.

TABLE 8. PRECISION AND RECOVERY DATA IN AQUEOUS MATRICES DRINKING WATER

	Sample	Low	Average High				Average	1		
Elem	ent Concn.	Spile	Recovery	S(FI)	RPD	Spiles	Recovery		RPO	
	(pg/L)	(µg/L)	R (%)			(µg/L)	R (%)			
Al	175	50	115.8	5.9	0.4	200	102.7	1.6	1.1	
Sb	<0.4	10	99.1	0.7	2.0	100	100.8	0.7	2.0	
e A	<1.4	50	99.7	8.0	2.2	200	102.5	1.1	2.9	
Ва	43.8	50	94.8	3.9	5.8	200	95.6	8.0	1.7	
Be	<0.3	10	113.5	0.4	0.9	100	111.0	0.7	1.8	
Cd	<0.5	10	97.0	28	8.3	100	101.5	0.4	1.0	
Cr	<0.9	10	111.0	3.5	9.0	100	99.5	0.1	0.2	
Co	0.11	10	94.4	0.4	1.1	100	93.6	0.5	1.4	
Cu	3.6	10	101.8	8.8	17.4	100	91.6	0.3	0.3	
Pb	0.87	10	97.8	2.0	2.8	100	99.0	0.8	2.2	
Mn	0.96	10	96.9	1.8	4.7	100	95.8	0.6	1.8	
Mo	1.9	10	99.4	1.6	3.4	100	98.6	0.4	1.0	
Ni	1.9	10	100.2	5.7 '	13.5	100	95.2	0.5	1.3	
Se	<7.9	50	99.0	1.8	5.3	200	93.5	3.5	10.7	
Ag	<0.1	50	100.7	1.5	4.2	200	99.0	0.4	1.0	
TI	<0.3	10	97.5	0.4	1.0	100	98.5	1.7	4.9	

TABLE 8. PRECISION AND ACCURACY DATA IN AQUEOUS MATRICES (Cont). WELL WATER

7724 TV - E11											
	Sample	Low	Average			High	Average	t			
Elem	entConcr.	Spike	Recovery	S(R)	RPD	Spike	Recovery	S(R)	RPO		
	(UO/L) (po/L)	R (%)		(ug/L)	R (%)				
Al	34.3	50	100.1	3.9	8.0	200	102.6	1.1	1.3		
Sb	0.46	10	98.4	0.9	1.9	100	102.5	0.7	1.9		
As	<1.4	50	110.0	6.4	16.4	200	101.3	0.2	· 0.5		
Ba	106	50	95.4	3.9	3.3	200	104.9	1.0	1.6		
Be	<0.3	10	104.5	0.4	1.0	100	101.4	1.2	3.3		
Cd	1.6	10	88.6	1.7	3.8	100	98.6	0.6	1.6		
Cr	<0.9	10	111.0	0.0	0.0	100	103.5	0.4	1.0		
Co	2.4	10	100.6	1.0	1.6	100	104.1	0.4	1.0		
Cu	37.4	10	104.3	5.1	1.5	100	100.6	8.0	1.5		
Pb	3.5	10	95.2	2.5	1.5	100	99.5	1.4	3.9		
Mn	2770	10	•	•	1.8	100	•	•	0.7		
Mo	2.1	10	103.8	1.1	1.6	100	102.9	0.7	1.9		
Ni	11.4	10	116.5	6.3	6.5	100	99.6	0.3	0.0		
Se	<7.9	50	127.3	8.4	18.7	200	101.3	0.2	0.5		
Ag	<0.1	50	99.2	0.4	1.0	200	101.5	1.4	3.9		
TI	<0.3	10	93.9	0.1	0.0	100	100.4	1.8	5.0		
Th	<0.1	10	103.0	0.7	1.9	100	104.5	1.8	4.8		
υ	1.8	10	106.0	1.1	1.6	100	109.7	2.5	6.3		
٧	<2.5	50	105.3	0.8	2.1	200	105.8	0.2	0.5		
Zn	554	50	•	•	1.2	200	102.1	5.5	3.2		
S(R)	Standan	d devi	iation of pr	ercen	t reco	very.					

RPDRelative percent difference between duplicate spike determinations.

Sample concentration below established method detection limit.
*Spike concentration < 10% of sample background concentration.</p>

TABLE 8. PRECISION AND ACCURACY DATA IN AQUEOUS MATRICES (Cont). POND WATER

PUND WATER												
	Sample	Low	Average			High	Average					
Elem	ent Concr.	Spike	Recovery	S(R)	RPD	Spike	Recovery	S(R)	RPO			
	(vg/L	(ug/L)	R (%)			(yg/L)	R (%)					
Al	610	50	•	•	1.7	200	78.2	9.2	5.5			
Sb	<0.4	10	101.0	1.1	2.9	100	101.5	3.0	8.4			
As	<1.4	50	100.8	2.0	5.6	200	96.8	0.9	2.6			
Ba	28.7	50	102.1	1.8	2.4	200	102.9	3.7	9.0			
Be	<0.3	10	109.1	0.4	0.9	100	114.4	3.9	9.6			
Cd	<0.5	10	106.6	3.2	8.3	100	105.8	2.8	7.6			
Cr	2.0	10	107.0	1.0	1.6	100	100.0	1.4	3.9			
Co	0.79	10	101.6	1.1	2.7	100	101.7	1.8	4.9			
Cu	5.4	10	107.5	1.4	1.9	100	98.1	2.5	6.8			
Pb	1.9	10	108.4	1.5	3.2	100	106.1	0.0	0.0			
Mn	617	10	•	•	1.1	100	139.0	11.1	4.0			
Mo	0.98	10	104.2	1.4	3.5	100	104.0	2.1	5.7			

ICO Indomentian Alexander

Ni	2.5	10	102.0	2.3 4.7	100	102.5	2.1	5.7
Se	<7.9	50	102.7	5.6 15.4	200	105.5	1.4	3.8
Ag	0.12	50	102.5	0.8 2.1	200	105.2	2.7	7.1
Π	<0.3	10	108.5	3.2 8.3	100	105.0	2.8	7.6
Th	0.19	10	99.1	3.5 10.5	100	93.9	1.6	4.8
ป	0.30	10	107.0	2.8 7.3	100	107.2	1.8	4.7
V	3.5	50	96.1	5.2 14.2	200	101.5	0.2	0.5
Ζn	6.8	50	99.8	1.7 3.7	200	100.1	2.8	7.7
S(R)	Standard	devi	ation of p	ercent reco	very.			
RPD	Relative	CATCA	nt differen	ce hetumer	عكم الم	ate enik	ماماء د	ani_

RPDRelative percent difference between duplicate spike determinations.

<Sample concentration below established method detection limit.</p>
*Spike concentration <10% of sample background concentration.</p>

TABLE 8. PRECISION AND ACCURACY DATA IN AQUEOUS MATRICES (Cont). SEWAGE TREATMENT PRIMARY EFFLUENT

Sample Low Average High Average ElementConon. Spike Recovery S(R) RPD Solve Recovery S(R) RPD (µg/L) (µg/L) R (%) (JON) A(X) Al 1150 50 3.5 200 100.0 13.8 1.5 Sb 95.7 1.5 10 0.4 0.9 100 104.5 0.7 1.9 As <1.4 50 104.2 4.5 12.3 200 101.5 0.7 Ba 202 50 79.2 9.9 2.5 200 108.6 4.6 5.5 Вe <0.3 1.8 4.5 100 106.4 0.4 0.9 10 110.5 Cd 9.2 10 101.2 1.3 0.0 100 102.3 0.4 0.9 Cr 128 10 1.5 100 102.1 1.7 0.4 Co 13.4 95.1 2.7 10 2.2 100 99.1 1.1 2.7 Cu 171 10 2.4 100 105.2 7.1 0.7 Pb 17.8 95.7 3.8 10 1.1 100 102.7 1.1 2.5 Mn 199 10 1.5 100 103.4 2.1 Mo 136 10 1.4 100 105.7 2.4 Ni 84.0 10 88.4 16.3 4.1 100 98.0.9 0.0 Se <7.9 50 112.0 10.9 27.5 200 108.8 3.0 7.8 Ag 10.9 50 97.1 0.7 1.5 200 102.6 1.4 3.7 TI <0.3 10 97.5 0.4 1.0 100 102.0 0.0 0.0 Th 0.11 10 15.4 29.3 0.8 8.2 1.8 30.3 100 u 0.71 10 109.4 1.8 4.3 100 109.3 0.7 1.8 ٧ <2.5 90.9 50 0.9 0.6 200 99.4 2.1 6.0 Zn 163 50 85.8 3.3 0.5 200 102.0 1.5 1.9

RPDRelative percent difference between duplicate spike determinations.

S(R)Standard deviation of percent recovery.

<Sample concentration below established method detection limit.</p>
*Spike concentration <10% of sample background concentration.</p>

TABLE 8. PRECISION AND ACCURACY DATA IN AQUEOUS MATRICES (Cont). INDUSTRIAL EFFLUENT

	Sample	Low	Average	_	_	High	Average	+		
Elem	entConco.	Spike	Recovery	S(R)	RPD	Spile	Recovery		RPD	
	(na/r)	(vg/L)	R (%)			(DQ/L)	R (%)			
Al	44.7	50	98.8	8.7	5.7	200	90.4	2.1	2.2	
Sb:	2990	10	•	•	0.3	100	•	•	0.0	
As	<1.4	50	75.1	1.8	6.7	200	75.0	0.0	0.0	
Ba	100	50	96.7	5.5	3.4	200	102.9	1.1	0.7	
Вe	<0.3	10	103.5	1.8	4.8	100	100.0	0.0	0.0	
Cd	10.1	10	106.5	4.4	2.4	100	97.4	1.1	2.8	
Cr	171	10	•	•	0.0	100	127.7	2.4	1.7	
Co	1.3	10	90.5	3.2	8.7	100	90.5	0.4	1.3	

0.9 100 92.5 2.0 1.6 Cu 101 10 2.6 100 Pb 294 108.4 2.1 10 2.8 100 103.6 3.7 Mn 154 1.6 10 Mo1370 10 1.4 100 0.7 107.4 7.4 5.0 100 88.2 0.7 1.0 Ni 17.3 10 129.5 9.3 15.1 200 Se 15.0 50 118.3 1.9 3.6 200 87.0 4.9 16.1 <0.1 50 91.8 0.6 1.7 Ag 90.5 1.8 5.5 100 Π < 0.3 10 98.3 1.0 2.8 109.6 1.2 2.7 100 108.7 0.0 0.0 Th 0.29 10 U 0.17 10 104.8 2.5 6.6 100 109.3 0.4 0.9 ٧ <2.5 74.9 200 72.0 0.0 0.0 50 0.1 0.3 Zπ 43.4 50 85.0 4.0 0.6 200 97.6 1.0 0.4 S(R)Standard deviation of percent recovery.

RPDRelative percent difference between duplicate spike determinations.

<Sample concentration below established method detection limit.</p>
*Spike concentration <10% of sample background concentration.</p>

TABLE 9. PRECISION AND ACCURACY DATA IN SOLID MATRICES EPA HAZARDOUS SOIL #884

	Sample	Low	Average			High	Average)	
Elem	entConcs.	Spike	Recovery	S(R)	RPD	Soke	Recovery	6(R)	RPO
	(mg/kg)	(mg/kg)	R (%)			(mg/kg)	- R (%)		
Al 8	5170	20	•	•	_	100	. •	•	-
Sb	5.4	20	69.8	2.5	4.7	100	70.4	1.8	6.5
As	8.8	20	104.7	5.4	9.1	100	102.2	2.2	5.4
Ba	113	20	54.9	63.6	18.6		91.0	9.8	0.5
Be	0.6	20	100.1	0.6	1.5			0.4	1.0
Cd	1.8	20	97.3	1.0	1.4		101.7	0.4	1.0
Cr	83.5	20	86.7	16.1	8.3		105.5		
Co	7.1	20	8.88	1.2	1.9		102.9	0.7	1.8
Cu	115	20	86.3	13.8			102.5		_
Pb	152	20	85.0	45.0	13.9				
Mn	370	20	•	•	12.7		85.2		
Mo	4.8	20	95.4	1.5	2.9	100	95.2		
Ni	19.2	20	101.7	3.8	1.0	100	102.3	0.8	8.0
Se	<3.2	20	79.5	7.4	26.4	100	100.7		26.5
Ag	1.1	20	96.1	0.6	0.5	100	94.8	8.0	2.3
TI	0.24	20	94.3	1.1	3.1	100	97.9	1.0	2.9
Th	1.0	20	69.8	0.6	1.3	100	76.0	2.2	
U	1.1	20	100.1	0.2	0.0	100	102.9	0.0	0.0
٧	17.8	20	109.2	4.2	2.3	100	106.7	1.3	2.4
Zn	128	20	87.0	27.7		100	113.4	12.9	14,1
S(R)	Standar	d devi	ation of p	ercen	t reco	very.			

RPDRelative percent difference between duplicate spike determinations.

<Sample concentration below established method detection limit.</p>
*Spike concentration <10% of sample background concentration.</p>
-Not determined.

+Equivalent



TABLE 9. PRECISION AND ACCURACY DATA IN SOLID MATRICES (Cont).

NBS 1645 RIVER SEDIMENT

	Sample	Low	Average			High	Averag	_		
Eie	ementConco.			S(R)	RPO	Soke	Recover		RPD	
	(mp/kg)		A (%)			(mg/kg)				
AI		20	•	•	_	100	•	•	_	
St		20	73.9	6.5	9.3		81.2	1.5	3.9	
As		20	104.3	13.0	7.6					
Ba		20	105.6	4.9	2.8				3.9	
Be		-	88.8	0.2					0.2	
Co		20	92.9	0.4	0.0	100				
	29100	20	•	•	_	100	•	•	_	
Co		20	97.6	1.3	26	100	103.1	0.0	0.0	
Cu		20	121.0	0.1	1.5	100	105.2			
Pb		20	•	•	-	100		_		
Мп		20	•	•	_	100	_	_	_	
Ma		20	89.8	8.1	12.0	100	98.4	07	0.0	
NI	41.8	20	103.7		4.8	100	102.2			
Se		20	108.3	14.3		100	93.9			
Ag	1.8	20	94.8		4.3	100	96.2			
TI	1.2	20	91.2			100	94.4		1.3	
Th	0.90		91.3	0.9	2.6	100	92.3		2.8	
U	0.79	20	95.6	1.8	5.0	100	98.5			
V		20	91.8	4.6		100		0.6	0.8	,
	1780	20	•	•	_	100	•	•	V.Q	
S(R	Standard	devia	don of p	ercen	t reco	verv.			_	4

RPO Relative percent difference between duplicate spike determinations.

TABLE 9. PRECISION AND ACCURACY DATA IN SOLID MATRICES (Cont).

EPA ELECTROPLATING SLUDGE #286

	Samola	سہ ا	Average	-			- CE 7200	
57~						High	Average	
	mentConon.			S(FI)	RPO	Solve	Recovery S(R)	RР
	(mg/ ng)		R (%)			(mg/kg)	R (%)	
	5110	20	•	•	_	100	• •	_
Sb	8.4	20	55.4	1.5	4.1	100	61.0 0.2	٥.
eA	41.8	20	91.0	2.3	1.7	_	94.2 0.8	
Ba	27.3	20	1.8	7.1	B.3	100		10.
Be	0.25	20	92.0	0.9			93.4 0.3	
Cd	112	20	85.0	5.2		100	88.5 0.8	
Cr	7980	20	• '	•	_	100	• • • •	_
Co	4.1	20	89.2	1.8	4.6	100	88.7 1.5	4.
Cu	740	20	•	•	6.0	100	61.7 20.4	
Pb	1480	20	•	•	-	100	* *	J.
Μn	295	20	•	•	_	100		_
Мо	13.3	20	82.9	1.2	1.3	100	89.2 0.4	1.0
Ni	450	20	•	•	6.8	100	83.0 10.0	
Se	3.5	20	89.7	3.7	4.2	100		4.5
٨g	5.9	20	89.8	2.1	4.6	100	91.0 6.0	
π	1.9	20	96.9	0.9			85.1 0.4	1.1
Πh	3.6	20			2.4	100	98.9 0.9	2.4
			91.5	1.3		100	97.4 0.7	2.0
J	2.4	20	107.7	2.0	4.6	100	109.6 0.7	1.8
1	21.1	20	105.6	1.8	2.1	100	97.4 1.1	2.5
	3300	20	•	•	_	100	•	_
(R)	Standard	devia	tion of pa	eceni	reco	verv.		_
RPD	Relative		nt differen	ace be			4	_

PD Relative percent difference between duplicate spike deter-

- < Sample concentration below established method detection limit.
- *Spike concentration <10% of sample background concentration.
- Not determined.
- + Equivalent

< Sample concentration below established method detection limit.

^{*}Spike concentration <10% of sample background concentration.

⁻ Not determined.

⁺Equivalent

APPENDIX E.2
IEA, INC.

SAMPLE CUSTODY

Date: 01/03/92 Page 11 of 61

D. Sample Custody

Due to the critical nature of the samples analyzed, IEA maintains strict security within the laboratory. Entrances to the laboratory are secured through the use of an electronic card access system. Visitors to IEA must enter through the lobby and sign in at the reception desk. Visitors to the office and/or laboratory must be accompanied by an employee at all times.

Samples are received in the shipping receiving department by the sample custodian or by an authorized member of the department. Upon receipt, the shipping container and the individual sample containers are inspected for damage. If any damage is present, a note is made in the project file, and the project manager or customer service department is notified. All sample information supplied by the client is reviewed and checked against the samples received. The number and type of samples received and the identity tags/labels are checked against the information supplied.

Each sample is assigned an IEA sample number. The IEA sample number is a combination of the IEA Client Number, IEA Client Project Number and the Sample Sequence Number.

Example: Sample number 789-100-2 refers to the second sample in the one hundredth project submitted by IEA client 789.

Each container is labelled with the assigned IEA sample number. If multiple containers are received for a single sample a unique alpha character is added to the end of the sample number assigned to each container. This practice allows each analysis to be traced to a single container.

Date: 01/03/92 Page 12 of 61

Each sample received is listed in the IEA Sample Check-In Log with the IEA sample number, client ID number, a complete description of each sample received, sample condition at the time of receipt, date of receipt, sample numbers or identifiers and any problems encountered in the course of receiving the samples. The receipt of chain-of-custody records (Attachment 1/SectionD) with the sample shipment is also noted on the check-in log.

A Project Data Sheet (Attachment 2/Section D) is completed for each set of samples received. This form serves as the primary source of information for the laboratory. The number and type of samples and sample containers received for the project are listed on the Project Data Sheet as well as type of analysis required, type of report required, turn around time and degree of chain-of-custody documentation required.

In-lab chain-of-custody records (Attachment 3/Section D) are maintained for each sample when requested by the client. For these samples, the in-lab chain-of-custody record is initiated upon sample receipt. Each movement of a sample or sample extract container into and out of the locked refrigerator system is recorded with date, time, bottle number, action (check in or check out), and signature of the individual accepting or relinquishing responsibility of the sample. The chain-of-custody records are kept in the associated project folder.

After receipt, samples are housed in lockable refrigerators. Samples are removed from the refrigerators by authorized employees for analysis and returned to the locked refrigerator system after completion of the analysis. Throughout the analytical process, each sample is either in the possession of authorized laboratory personnel or secured in a lockable refrigerator inside the secured laboratory area.

Analytical data reports are kept in filing cabinets which are locked at the end of each business day. Sensitive documents are shredded prior to disposal.

In situations where IEA is requested to send sample containers out of our facility, we use a cooler known as a Transpak. This is a corrugated cardboard box lined with an insulated

Date: 01/03/92 Page 13 of 61

insert. Foam packaging in the inserts preclude container movement and breakage as the holes are cut specifically for protocol-required containers. These Transpaks come in several sizes, and they can be requested by contacting the Client Services Department of IEA - North Carolina. Sampler instructions (Attachment 4/Section D) are included with each Transpak.

Date: 01/03/92 Page 14 of 61

Attachment 1 / Section D



CHAIN OF CUSTODY RECORD

	REGULATORY CLA	SSIFICAT	ION - PLEASE SPECIFY
□ NPDES	☐ DRINKING WATER	RCRA	☐ OTHER



PROJE	CT#				PROJ	ECT NAM	4E		c		7011					REQL	JESTE	ED PA	RAMI	ETER	S			
		,							# T	MA	TRIX	$\overline{}$		7	7					$\overline{}$	$\overline{\mathcal{I}}$			
SAMPLER	S: (SIGN	ATURE)							OF I	s	w	ĺ					Ź					<i>i</i>		
									N E R S	0	WATER						/					/	1	/
SAMPLE I.D.	DATE	TIME	COMP	GRAB		STATIC	N LOCA	TION	S		Ř	/ 			<i></i>	<u> </u>			/		<i> </i>	L	/ /	<u>/</u>
																	_							
		-														 				<u> </u>				
										-				 	 									
		;										·········	1											
											+				-		<u> </u>							
						······				-				 								<u> </u>		
										_	-		 			-			ļ					
25, 110, 11	0.120.0	(401011			1	T11.45		DE0511	(50.0)			-	711.45	R							0.50	DUCI	2/0	
RELINQUI	PHED R.	Y (SIGNA	URL	=) D	ATE	TIME		RECEIV	FD R.		DAT	=	TIME		U	EA QUO	JIE N). 			IEA	RUSH	NO.	
RELINQUI	SHED B	Y (SIGNA	TURI	E) D	ATE	TIME	RE	CEIVED F	OR LAB BY	,	DAT	E	TIME	PRO	JECT	MANAC	GER (P	LEASE P	RINT)			P.O. NO).	
						IEA REN	AADKS								X		 	CICI	D REMA	DKS				
						ICA NEN	CANA											FIEL	Y LICIMA	AUV 2	· · · · ·			 .

Date: 01/03/92 Page 15 of 61

Attachment 2 / Section D



DATA FOR PROJECT NO.

N. B. I. A.	CUE	NT REPRESENTATIVE		2(2'50'5)&	PF	OJECT I.D. 1
		(ADDRESS ⊅(C. 5)			PURCH	ASE ORDER NO.
						NG ADDRESS
	•				☐ AS LISTED	☐ OTHER (See Below
ash #	Client I.D.	Sampling Date	Job Codes	Test Code	es Bottle Size	Location
				+		
					,	
					`	
		-				
-						
				1		
				-		
			-			
		COMMENTS			TURNAROUN	D REQUIREMENTS
					WORK D	OF PRICING PACTOR
		•			NORMAL 15	5 1.0 X
					RUSH	
Date S	amples Received	Report Du	e Date	L	ab Due Date	Carrier

(1) amples received after 2:00 pm will be assigned the date of the following workday.
 (2) epresents the date that results are shipped to the customer.
 (3) __xcludes weekends and holidays.

Date: 01/03/92 Page 16 of 61

Attachment 3 / Section D

		San	nple Manag	gement Cha	ain of Cus	tody				
EPA ID:			Matri	x: S/W	IEA ID:					
Log-In By	:		Date:							
	S	SAMPLE II	SAMPLE OUT							
Bottle	Date	Time	Code	Init.	Date	Time	Location	I		
						,				
	<u> </u>									
					· · · · · · · · · · · · · · · · · · ·			 		
		į '	1	1 1				i		

Bottles letters available:	
Applicable codes are:	EX = Extraction TR = Transfer DI = Dispose
	ST = Storage
Verified by:	
Init.	Date

GC/MS Chain of Custody

	S	SAMPLE II	SAMPLE OUT					
Bottle	Date	Time	Code	Init.	Date	Time	Location	Init.
						`		
	i							
								

Date: 01/03/92 Page 17 of 61

Attachment 4 / Section D

Date: 01/03/92 Page 18 of 61

ATTENTION!

SAMPLE INSTRUCTIONS

PLEASE FILL EVERY BOTTLE PROVIDED. IF THIS IS NOT POSSIBLE, IMMEDIATELY CONTACT IEA'S CLIENT SERVICES DEPARTMENT AT (919) 677-0090 IN ORDER TO AVOID UNNECESSARY DELAYS IN ANALYSIS.

ALL SAMPLES SHOULD BE RECEIVED WITHIN 24 HOURS OF SAMPLING. SAMPLES RECEIVED AFTER 24 HOURS FROM SAMPLING MAY REQUIRE ACCELERATED TURNAROUND IN ORDER TO MEET PROTOCOL HOLDING TIME REQUIREMENTS.

PLEASE RETURN COOLANT PACKS WITH THE TRANSPAK.

Date: 01/03/92 Page 19 of 61

SAMPLER INSTRUCTIONS

This sample package has been prepared for you with the objective of helping to maintain the integrity of your samples. It is therefore vital that you read and follow these instructions.

- 1. Carefully open the sample package and check the contents. If any bottles are missing, broken, or damaged, call the laboratory immediately at 919-677-0090.
- 2. Remove and freeze the freezer packs included with the shipping container for at least eight hours. They must be solidly frozen upon packing the samples for return shipment. The freezer packs will maintain a sufficiently cool temperature for approximately 72 hours.
- 3. Note the following before sampling:

3.1 40ml Volatile Vials

40ml volatile vials must not contain any air bubbles. Fill the vial to just below the point of overflow, until there is a convex meniscus (see picture at the left). Carefully slide the teflon insert over the meniscus, teflon (stiff) side down (against the sample). Screw the cap on the vial, and check for air bubbles. If air bubbles are present, repeat the capping procedure, or draw another sample, if necessary. Volatile bottles do not normally contain preservative chemicals.

3.2 **Bacteria Sampling Bottles**

Handle sterile bacteria sampling bottles carefully to avoid contamination. Do not open the bottles until ready to sample. Fill to within half an inch of the top, and tighten the cap securely.

Date: 01/03/92 Page 20 of 61

3.3 Other Sampling Bottles

Some sample bottles contain strong acids or bases as preservatives. These bottles have color coded cautionary labels. Handle with care. Do not prerinse or overfill bottles having color coded cautionary labels. Tighten cap securely when filled. The color code used is as follows:

Red: Yellow: Preserved with nitric acid Preserved with sulfuric acid

Blue:

Preserved with hydrochloric acid

White:

No preservative

Green:

Preserved with sodium hydroxide

(basic) solution

- 4. Complete the sample tags and labels by filling in the sample I.D., sampling address, the sampling point, date and time (24 hr. format; for example: 8:00 am = 0800 hours or 10:00 pm = 2100 hours). Indicate if the sample is a grab or composite. The sampler should initial at the appropriate space.
- 5. Make sure all caps are secure, and attach labels and tags to correct bottles. Repack the samples for return shipment to the laboratory, making sure to include the freezer packs. Ship by a route which will ensure delivery within 72 hours.
- 6. If you have any questions, call IEA's sample receiving department, or our client representative, at 919-677-0090 between 8:00 am and 5:00 pm Monday through Friday.

WARNING!!!

40ML VIALS FOR

VOLATILE ANALYSES

(602, 8020, 624 or 8240)

ARE PRESERVED WITH

CONCENTRATED HCL.

PLEASE USE CAUTION

WHEN HANDLING THESE

VIALS.

APPENDIX E.3

IEA, INC.

DATA REDUCTION AND REPORTING

Date: 01/03/92 Page 29 of 61

Data Reduction and Reporting

The data associated with each analysis are hardcopied for permanent storage either through the printing of computer files or through hand entry into bound laboratory notebooks. All notebook entries are dated and signed by the analyst. Standardized notebook and logbook requirements include the following:

- (01)Preprinted pages
- (02)Prenumbered pages
- (03)Bound logbooks
- (04)Document controlling of logbooks
- (05)Archival of old logbooks
- (06)Acceptance criteria in logbook
- (07)Making corrections
- (80)Secondary review of logbook entries

Notebook entries, or any other general laboratory records, must be made in blank ink. Any logbook or notebook entries that are corrected are made by using a one-line strikeout in black ink. All corrections are signed and dated.

Data reduction includes all processes that change either the form of expression (i.e., the units of measure) or the quantity of data values (rounding). It often involves statistical and mathematical analysis of data and usually results in a reduced subset of the original data set. Data reduction is performed either manually by the analyst or by computer systems interfaced to the analytical instruments. Whenever such procedures are employed within the laboratory Network, mathematical procedures have been verified for accuracy of computation.

All data are subjected to a multilevel review. All data reports are reviewed by the department supervisor prior to release for final report generation. A cross section of data reports are reviewed by the Laboratory Director. All final data reports are reviewed by a member of the senior technical staff prior to release to the client. It is the responsibility of the Quality Assurance Manager to review a random sample of five percent of final reports

Date: 01/03/92 Page 30 of 61

prior to shipment. The members of the senior technical staff and Quality Assurance are not members of the analytical production laboratories.

Out-of-control conditions identified by the analyst, supervisor, manager or technical staff member are investigated, corrected and documented. Out-of-control conditions which are caused by the sample itself, are addressed in a project narrative in the final report.

All elements of the IEA-North Carolina Quality Assurance Program must be satisfied before a data report may be released to the client.

APPENDIX E.4

IEA, INC.

PERFORMANCE AND SYSTEM AUDITS

Date: 01/03/92 Page 31 of 61

I. **Performance and System Audits**

Each quarter the overall performance of the laboratory staff is evaluated and compared to the performance criteria outlined in the quality assurance manual and the standard operating procedures. The Quality Assurance Manager conducts a laboratory audit to evaluate the performance of the laboratory staff and compares that performance to the requirements of the quality assurance program. During this process, the records, standard operating procedures and adherence to those standard operating procedures are examined. The results of the audit process are summarized and issued to each department supervisor and the Laboratory Director.

Known intralaboratory performance samples are analyzed in the form of sample spikes, duplicates and duplicate sample spikes on a continuing basis. Two (2) such samples are processed for every twenty (20) production samples.

"Blind" intralaboratory performance audits are conducted monthly. Samples containing known analyte concentrations are introduced into the laboratory as client samples. These samples are analyzed and reported in the same manner as normal production samples. The results and the true values of each sample are reported to the department supervisors and the Laboratory Director upon receipt of the data by the Quality Assurance Manager.

IEA participates in interlaboratory performance audits through the various state and federal certification programs. IEA is an active participant in the U.S. Environmental Protection Agency's Contract Laboratory Program (CLP) and the U.S. Army Corps of Engineers accreditation program. A list of IEA-North Carolina certifications, as well as those for our other six network laboratory operations, summarizes our analytical capabilities (Attachment 1/Section I).

Date: 01/03/92 Page 32 of 61

Attachment 1 / Section I

Date: 01/03/92 Page 33 of 61

STATE CERTIFICATIONS

Certifying State	Program Type	NC	MA	VΤ	FL.	СТ	ĮŊ	n
Alabama	DW	Y						
Arkansas	None	-					_	
Connecticut	General		- -		-	<u> </u>	Y	Y
California	DW				 	 	 	+
Cantomia	ww				 		 	╁
	HW				 	 		
Colorado	DW		<u> </u>		 			
Delaware	None	-		_	<u> </u>	<u> </u>		
Florida	DW	Y			Y		 	╁╌
ji i	ww				Y		 	╁
Georgia	DW	Y			 		 -	╁
Illinois	DW						 	Y
Indiana	None				- -		-	+
Kentucky	?	•	ļ <u>.</u>			 		
Louisiana	None				 			
Main	DW	-	- Y	Y			 	
Massachusetta	DW	Y	Y	Y		Y	 	┼
Massachuseus	ww	Y	Y			- <u>'</u>		<u> </u>
Minnesota		T .	T				 -	
Missouri				· · · · · · · · · · · · · · · · · · ·	 	 	 	
New Hampshire	DW		Y				 -	-
ivem transfame	ww		Y		 			
New Jersey	DW	Y	1			Y	Y	╂
Mem Tetsea	- ww	Y				<u>,</u>	Y	
New York	DW	- 1			 	Y	Y	
IN TOR	ww					Y	Y	┼──
	HW					Y	Y	
North Carolina	DW	- V				<u> </u>		
	ww	Y			 	<u> </u>		╂
0		Y	Y		 			
Pennsylvania Rhode Island	DW None	-		_	_		Y -	
South Carolina	DW	Y			 		Y	
South Carolina	ww	Y					Y	
Теплемесе	DW	Y					Y	
Texas	General	1					- I	┼──
								
Vermont	DW			Y				
Virginia	DW	Y						├
	ww	Y			 		_	
Wisconsin	General						l	Y

Date: 01/03/92 Page 34 of 61

Footnotes:

-Drinking Water certification program DW

-Wastewater certification program ww

HW -Hazardous Waste certification program

Y -Laboratory has some form of certification under the specific program.

None -No program currently exists in this State, therefore, certification is not available.

APPENDIX E.5
IEA, INC.
CORRECTIVE ACTION

Date: 01/03/92 Page 41 of 61

L. **Corrective Action**

Corrective actions can be initiated at several operational levels; however, they always involve QA personnel. In each case, after an assessment of the situation, appropriate steps are taken to correct the problem. Depending on the severity of the problem, corrective actions may be taken at the analyst level, department level, or within the entire laboratory. IEA recognizes the importance of corrective action to maintain a high quality program. In this light, all data are reviewed for completeness, accuracy, and compliance with QC criteria both within the analytical laboratory by peer review and by the department supervisor or manager.

In general, there are three major types of corrective actions which may be initiated at IEA:

Sample Problems

Individual samples or matrix problems are usually handled within the analytical laboratory. Corrective actions may include complete reextraction, repreparation, analysis, clean-up, dilutions or matrix modifications. All actions taken are documented with the analytical results.

OC Batch Problems

An entire batch of samples may require corrective action if QC criteria are not met. Department managers and QA staff are involved in the decisions for actions which include reanalysis, reextraction, etc. The QA department personnel review both sets of data where applicable to determine if the problems have been resolved.

Systematic Problems

Those problems of a procedural nature are handled by the laboratory managers and QA manager. For major operational changes, initiation of such are made only after approval by the QA manager and the Laboratory Director.

APPENDIX F

FIELD EQUIPMENT
STANDARD OPERATING PROCEDURES

APPENDIX F

TABLE OF CONTENTS

- F.1 Field Measurements of pH
- F.2 Field Measurements of Specific Conductance and Temperature

APPENDIX F.1 FIELD MEASUREMENTS OF pH

Field Measurements of pH

<u>Method</u>: Electrometric measurement of pH

Sensitivity: 0.1 pH Unit

Optimum Range: 1 - 12 pH Units

Sample Measurement: On-site upon sample collection

Reagents and Apparatus:

1. pH meter (Orien Model 211 min-pH meter or equivalent)

- 2. Combination electrodes
- 3. Beakers or plastic cups
- 4. pH buffer solutions, pH 4, 7, and 10
- 5. Deionized water in squirt bottle
- 6. All glassware shall be soap and water washed, followed by two hot water rinses and two deignized water rinses.

Procedures

A.Calibration:

- 1. Place electrode in pH 7 buffer solution.
- 2. After allowing several minutes for meter to stabilize, turn calibration dial until a reading of 7.00 is obtained.
- 3. Rinse electrode with deionized water and place in pH 4 or pH 10 buffer solution.

NOTE: When calibrating the meter for samples with pH < 8.00, use buffers 7 and 4; and, for samples with pH > 8.00, use buffers pH 7 and 10.

- 4. Wait several minutes and then turn slope adjustment dial until a reading of 4.00 or 10.00 is obtained.
- 5. Rinse electrode with deionized water and place in buffer pH 7. If meter

reading is not 7.00, repeat steps 2 - 5.

B. Sample Measurements:

- 1. Calibrate the meter according the calibration procedure.
- 2. Pour sample into a clean beaker or plastic cup.
- 3. Place the electrode in the sample solution. Make sure the white KCl junction on side of electrode is in solution. The level of electrode solution shall be one inch above sample to be measured.
- 4. Record the reading along with the temperature of the solution in the field/laboratory logbook.
- 5. Rinse electrode with deignized water between samples. Recheck calibration with pH 7 buffer solution after every 5 samples.
- 6. Repeat step 2-5 for each sample.

Quality Control:

- 1. Recheck calibration with pH 7 buffer solution after every 5 samples. The reading shall not exceed 7.00 +0.01 pH unit.
- 2. If, during meter calibration or calibration check, the meter fail to read 4.00 or 10.00, something may be wrong with the electrode. The cause shall be investigated and corrected. If problem lies in the electrode, the electrode shall be replaced.
- 3. pH is temperature dependent analysis. Therefore the temperature of buffer and smaples shall be recorded. The difference of temperature between the buffer solutions and samples shall be within about 20°C. For refrigerated or cooled samples, refrigerated or cooled buffer solutions shall be used to calibrate the meter.
- 4. When not in use, the electrodes shall be stored in pH 4 buffer.
- 5. Weak organic acids, inorganic salts, and oil and grease interferes the pH measurements. If oil and grease are visible, note it on the field logbook and data sheet. Clean the electrode with soap and water,, followed with 10% HCl. Then recalibrate the meter.
- 6. Before going into the field:

The following preparations shall be performed:

a. Check batteries:

- b. Calibration at pH 7 and 4 to check the electrode.
- c. Obtain fresh buffer solutions.
- 7. Following field measurements:
 - a. Report any problems;
 - b. compare with previous data;
 - c. Clean all dirt off meter and inside case;
 - d. Make sure electrode is stored in pH 4 buffer.

References:

1. EPA Method No.150.1, "Methods for Chemical Analysis of Water and Wastes" 1979, Revised 1983.

> TOTAL P.06 P.06

APPENDIX F.2

FIELD MEASUREMENTS OF SPECIFIC CONDUCTANCE AND TEMPERATURE

Field Measurements of Specific Conductance and Temperature

Method: Specific Conductance, umhos at 25°C

Detection Limits: 1 umho/cm at 25°C

Octimum Ranges: 0.1 - 100,000 umhos/cm

Sample Measurement: Measure on-site upon sample collection

Resources and Apparatus:

1. Conductivity meter (YSI or equivalent) and electrodes

2. Deionized water in squirt bottle.

- 3. Stock potassium chloride soultion, 1.00 N: Dissolve 74.555 g of KCl in Milli-Q water and dilute to 1,000 ml in a 1-liter volumetric flask.
- 4. Standard potassium chloride solution, 0.0100 N: Pipet 10 ml of stock solution into 1,000 ml volumetric flask and dilute with milli-Q water to the mark.

Procedures:

- 1. With mode switch at off position, check meter zero. If it can not be zero, use meter screw and adjust to zero.
- 2. Plug probe into jack on side of meter.
- 3. Turn mode switch to red line, and turn red line knob until need aligns with red line on dial. Change batteries if it can not be aligned.
- 4. Totally immerse probe in sample. Note: Do NOT allow the probe to touch the sample container.
- 5. Turn mode switch to appropriate conductivity scale, X100, X10, or X1. Use a scale that will give a mid-range output on the meter.
- 6. Wait for the needle to stabilize (about 15 seconds) and record conductivity multiplying by scale setting in field logbook.
- 7. While gently agitating the probe, take sample temperature (OC) and record in field logbook.

- 8. Rinse probe with deionized water.
- 9. Calculate specific conductivity using the following equation:

$$G_{25} = \frac{G_{T}}{\{1+0.02 (T-25)\}}$$

Where:

 $G_{25} = \text{conductivity at } 25^{\circ}\text{C}, \text{ umhos/cm}$

T = Temperature of sample, °C

GT = Conductivity of sample at temperature TOC, umhos/cm

- 10. Record specific conductivity and temperature in the field logbook, and on appropriate data sheet. Report results for the standard solution with each data set.
- 11. Record on field logbook which meter and probe were used.

Quality Control:

- 1. After use, the meter must be wiped clean as possible.
- 2. After returning to the laboratory, compare results with previous data, and report any problems encountered to the lab personnel.
- 3. Recheck calibration after every 5 samples.

References:

- 1. EPA Method No.120.1, "Methods for Chemical Analysis of Water and Wastes." 1979, revised 1983.
- 2. Standard Methods, 15th Edition.